

ELECTROCHEMICALLY INDUCED COLD FUSION

by

John N. Harb and William G. Pitt
Department of Chemical Engineering
Brigham Young University
Provo, UT

The following is in response to questions posed by the Panel of Cold Fusion of ERAB in a letter to Dr. Steven Jones from John R. Huizenga dated August 9, 1989.

PAST WORK

We have developed a beaker cells whose mass is isolated from the environment for use in our Cold Fusion experiments. This cell features a catalyst system to recombine evolved D_2 and O_2 . The recombination is expected to concentrate any helium and/or tritium produced by the fusion reaction. A gas collection system provides samples for chemical analysis. Experiments were conducted in the presence of a sensitive neutron detector and the electrolyte was analyzed for tritium. In addition, ICP analysis and SEM/EDX analysis were performed on the electrodes.

A rough schematic design of this cell is shown in Figure 1. The host metal electrode is a cylindrical palladium rod of 0.5 mm diameter. This rod is surrounded by a concentric counter electrode of palladium wire. The electrodes are contained in a glass cell surrounded by an evacuated glass jacket. Calibrations were performed to determine the heat loss from this cell. Calibrations showed that the heat sensitivity of the cell was about 0.25 watts.

The key feature of this cell is that mass inside the cell is completely isolated from the outside environment. This isolation is made possible through the use of a recombination chamber which provides a platinum catalyst on which the oxygen and deuterium (or hydrogen, in reference blank experiments) may recombine. Excessive pressure build up owing to O_2 and D_2 is thus eliminated. A cation exchange membrane is placed between the anode and cathode to prevent O_2 and D_2 from mixing inside the cell. The evolved gases exit the cell separately, pass through separate flow meters and then recombine in the recombination chamber. The volume of the recombined water is measured, and the water is cooled to room temperature before it is returned to the glass cell. The recombination is performed externally to the glass cell so that the heat evolved in recombination will not build up in the electrolyte.

Gases remaining in the cell and recombination chamber are collected and analyzed by mass spectrometry. After the experiments are finished, water from the recombination chamber and electrolyte are analyzed for tritium using beta counting on a Beckman Betamate instrument. Key features of the cell are summarized in the list that follows:

- 1) Separate electrode compartments eliminate in-cell recombination or burning of palladium.
- 2) The closed system with external recombination prevents escape of fusion products. Both gas and electrolytes are analyzed for fusion products.
- 3) Use of a palladium anode prevents contamination of the cathode with other metal cations.

- 4) Threemolar lithium deuterioxide is used to provide high conductance and thus low joule heating.
- 5) Four thermocouples in the cell measure electrolyte temperatures with an accuracy of about 0.1° centigrade.
- 6) The vacuum jacket around the cell minimizes heat loss and allows us to detect heat at the level of 0.25 watts.
- 7) Pressure, gas flow rates, temperatures, voltage, cell voltage, and cell current are continuously recorded on a data acquisition system. The experiments are performed at constant current.
- 8) The concentric geometry of the anode provides a uniform current density at the cathode.
- 9) The electrochemical cell is of the size to fit conveniently in the neutron spectrometer developed by Steve Jones, Bart Czirr and Gary Jensen of B.Y.U.

Neutron emission measurements were taken during the course of electrolysis of the D_2O in the cell. A total of 176 hours of foreground neutron emission measurements were made in two separate runs, the first of 56 hours, the second of 120 hours. A total of 139 hours of background neutron emissions were recorded during electrolysis of 3M lithium hydroxide in light water. In the heavy water runs, the single neutron emission events were about 5.9 standard deviations above light water background. The coincident neutron emission events, or events in which more than one simultaneous neutron capture were recorded were 3.4 standard deviations above background. No excess heat was determined during any of the foreground or background runs. No tritium above background tritium in the D_2O electrolyte was detected in any of the foreground or background experiments. No 3H or 4H was detected by mass spectrometry. SEM and EDX analysis of the cathode showed palladium dendrite formation covering the cathode with very small impurities of iron and copper. The anode was pure palladium. Several pits were formed in the anode during the course of the experiments.

PRESENT RESEARCH

We are continuing to use the same electrochemical cell to study neutron emissions during electrolysis of heavy water. In present experiments we are maintaining the cell at room temperature or colder through the use of cooling coils which pass through the electrolyte. In addition to palladium we are also using anodes of nickel and platinum in an attempt to keep some electrochemical cells running for more than two months. Our present efforts will continue to concentrate on measurement of neutron emissions and detection of tritium. In the long term experiments which run for more than two months, the cells are not placed in the neutron counter—they are analyzed daily for tritium production. The data acquisition system will indicate if any anomalous heat is produced. In the event that we detect anomalous heat or tritium, we will immediately place the cell on the neutron spectrometer to determine if neutron emissions are coincident with tritium and/or anomalous heat generation.

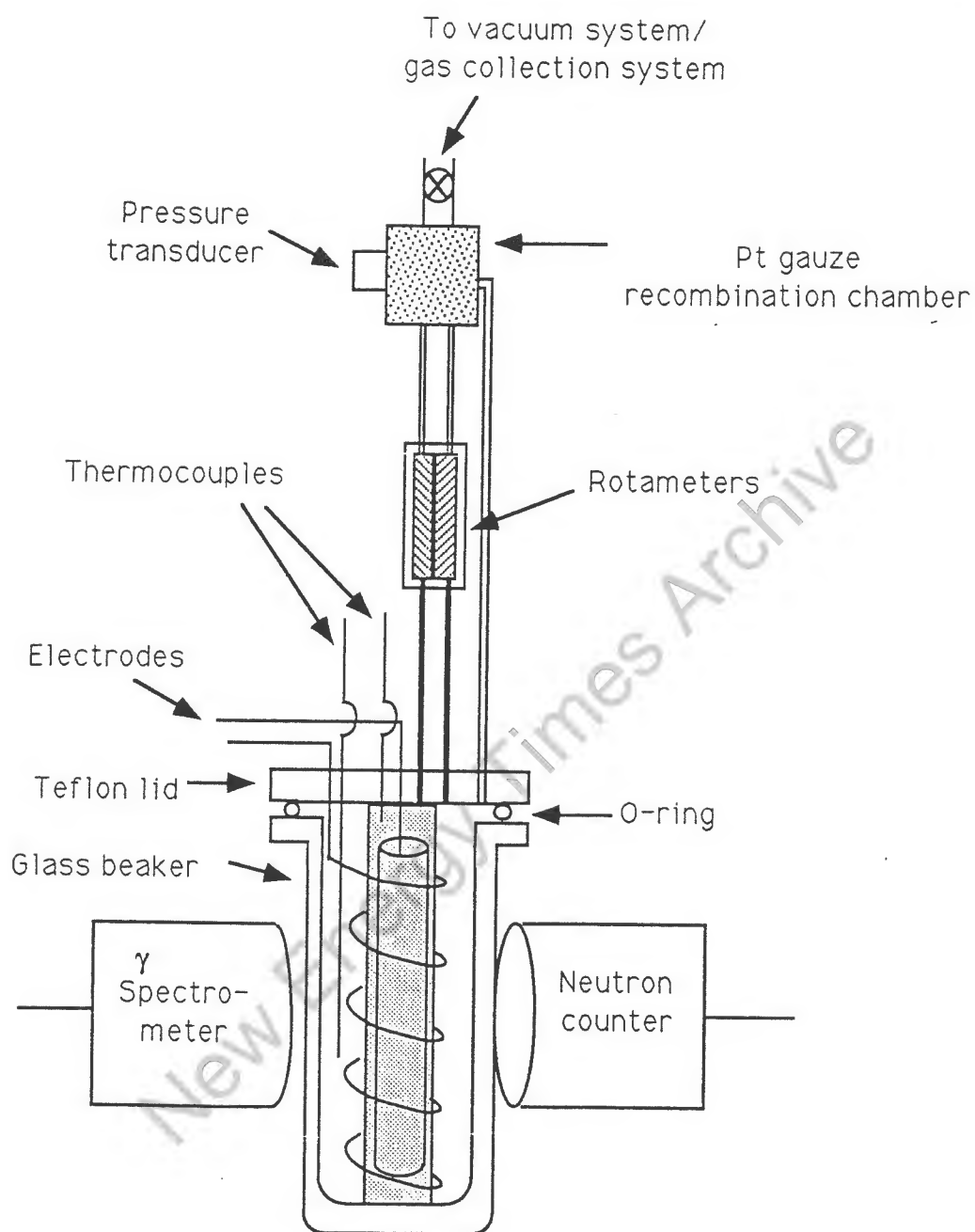


Figure 1. Schematic of Fusion Cell

A. Narath
President

Sandia National Laboratories

Albuquerque, New Mexico 87185

SEP 15 1989

Professor John R. Huizenga
Energy Research Advisory Board
c/o Dr. William Woodard
U.S. Department of Energy
ER-6, 3F-061
Office of Energy Research
1000 Independence Avenue, S.W.
Washington, DC 20077-9381

Dear Professor Huizenga:

Enclosed is the final report by Sandia National Laboratories to the ERAB Cold Fusion Panel. Our conclusions remain similar to the preliminary report you received in June.

We hope this information will prove useful to the Panel. If you have further questions please contact Tom Picraux at 505-844-7681 (Fax 846-2009).

Sincerely yours,



Copy to:

20 O. E. Jones
1000 V. Narayanamurti
1100 F. L. Vook
1110 S. T. Picraux
1800 R. J. Eagan
1840 R. E. Loehman
8340 W. Bauer

Information Copy to:

1090 J. E. Schirber
1111 B. L. Doyle
1111 J. A. Knapp
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1112 P. M. Richards
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1841 S. S. Tsao
1841 T. R. Guilinger
1841 B. D. Kay
2147 N. A. Godshall
6224 E. P. Roth
6233 W. H. Casey
8347 K. Wilson
9111 R. I. Ewing

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FINAL REPORT TO THE ERAB COLD FUSION PANEL
COLD FUSION RESEARCH RESULTS

Sandia National Laboratories

September 15, 1989

New Energy Times Archive

I. OVERVIEW

Since the announcement of cold fusion in late March by the University of Utah investigators we have carried out an extensive range of research activities at Sandia National Laboratories in an attempt to duplicate those experiments and also to gain evidence for the existence of this phenomenon. The program has focused on experiments of both the University of Utah type, where significant excess heat was reported, and on the Brigham Young University type, where a lower level of fusion reaction was reported. Approximately twenty members of technical staff have been involved, representing expertise in materials science, radiation physics, electrochemistry, solid state physics and metallurgy. We find no evidence for the existence of cold fusion.

II. DETECTION SYSTEMS

Three special detection capabilities have been utilized in these studies. First we have set up a multi-detector neutron detection system of ^3He proportional counters and located it underground on Kirtland Air Force Base near our Albuquerque location. This has allowed us to carry out studies with both electrochemical cells and high pressure gas cells with the capability of detecting continuous neutron emissions corresponding to production levels as low as 100 neutrons/hr or bursts as low as 50 neutrons. Not only have these underground experiments allowed us to reduce background levels by three orders of magnitude from aboveground tests, but by correlating the signals from three independent detectors operating simultaneously we have found that we are able to essentially eliminate spurious artifacts which occasionally give false signals in one detector system but not in all three simultaneously. Single-detector artifacts were observed that closely mimic both continuous and burst emissions reported previously as being due to cold fusion. We remain willing to test demonstrated electrolytic or gas cycling examples of cold fusion using our low background, multiple counter configuration.

A second special capability is a closed cycle Freon calorimeter system in which we are performing electrochemical cell experiments. This calorimeter has the advantage of being sensitive to the presence of small amounts of excess heat at high power levels (e.g., 1% at 20 W). Also, the total energy input is monitored from initiation of the experiment. We have conducted experiments with cast palladium electrode volumes $>0.5\text{ cm}^3$ at current densities above 300 mA/cm^2 for up to 30 days, while taking into account all experimental details proposed to be important by Pons and Fleischmann and by Huggins.

Finally we have utilized sensitive charged particle detection in conjunction with thin ($25\text{ }\mu\text{m}$) Pd foils where the foil forms one wall of the electrochemical cell. This approach allows the t-p branch of the d-d fusion reaction to be detected with essentially no background at very large detection efficiencies. The above three detection approaches have allowed us to exceed the sensitivity of the U. of Utah and the BYU experiments.

III. COLD FUSION EXPERIMENTS

A. Electrochemical Cells—(1) U. of Utah Type: Electrochemical cells designed after the Pons and Fleischmann experiment have been operated using various electrode preparations (including cast Pd, Pd 1 at.% Li alloy and t-loaded Ti films), current densities (up to 990 mA/cm^2), and times (up to 77 days). We have searched for the presence of radiation using neutron detectors, tritium by analyzing the electrolyte, and excess heat with our calorimetry system. (2) BYU Type: Electrochemical cell experiments of the type reported by Jones et al. have been conducted using a wide variety of electrodes including Ti and Pd, immersed in LiOD or the Jones "mother earth" electrolytes. These were carried out under normal laboratory conditions, as well as at the high neutron sensitivity underground location. (3) Thin Wall Cell Type: We have also utilized our thin wall cell configuration to obtain an order of magnitude higher sensitivity than in the Jones experiment (for bulk reaction rates within the Pd thin foils). These studies, as well as those of the BYU type, have been carried out for both steady-state and pulsed charging modes to look for cold fusion under highly non-equilibrium conditions.

B. Pressure/Temperature Gas Cycling—We have carried out a wide variety of experiments in which pressure and temperature have been cycled between 1 atm and 4 kbar and between room temperature and 77 K in deuterium gas. Materials included Pd powder, Ti powder, sintered Pd-Ti powder of the prescription given by Jones, TiD_2 , Ti electrodes soaked in Jones electrolyte, and TiV and Zr shavings. The sensitive underground neutron detection system was used.

C. Plasma Loading—We have conducted experiments in which the atomic deuterium was introduced into Pd powder by plasma loading and the radiation measured by neutron and charged

particle detectors. Experiments focused on understanding conditions for minimizing surface recombination and allowed loading ratios of about 0.9 D-Pd to be obtained.

D. Implantation Loading—Direct deuterium ion implantation into Pd and Zr at low substrate temperatures (40 K) was carried out to achieve superstoichiometric conditions. In the case of Pd, for example, we achieved loading ratios as high as D/Pd \approx 1.6; in all cases charged particle detectors were used to monitor the t+p branch of the cold fusion reaction.

E. Chemical Assays for Fusion Products—We have collaborated with Rockwell International (Canoga Park, CA) in the search for evidence of ^3He or ^4He fusion products in Pd electrodes from electrochemical cells provided by a number of U.S. laboratories. Presently we are in the process of participating in the double-blind helium tests being coordinated by Battelle-PNL on University of Utah samples. Measurements at Rockwell's Helium Assay Facility involve vaporizing samples under vacuum, passing all gases through multiple getter stages and measuring the helium concentrations using a precision mass spectrometer that had been calibrated with Pd samples implanted at various ^4He fluences by Sandia. The ultimate detection level is $\sim 1 \times 10^{10}$ helium (^3He or ^4He) atoms per gram of Pd.

In all the experiments which we have completed we have found no evidence for the presence of any radiation, radiation products, or excess heat which would have indicated the presence of cold fusion. We believe that we have faithfully reproduced the experimental conditions as best known and have a greater sensitivity for the presence of the phenomenon than that reported by the original investigators.

IV. SUPPORTING STUDIES

A. Loading Kinetics of Electrochemical Cells—Two experiments were conducted to better understand the actual loading kinetics of Pd electrodes, since it has been speculated that high D-to-metal loading ratios are important and there have been questions as to the loading times, cell operating conditions and electrolyte conditions under which proper loading may be achieved. In the first experiments thin wall cells were utilized in conjunction with an external ion beam for in situ analysis of the D concentration within the Pd metal during cell operation. Loading dynamics, as well as D/H poisoning kinetics, were examined. In the second set of experiments the permeation through electrochemical cell thin foils was used to examine the influence of palladium electrode surface preparation on the loading achieved.

B. Deuterium Atom Spacing—Molecular dynamics and total energy band structure calculations were carried out to examine the minimum D-D spacings which should be expected. Theoretical consideration was also given to possible closer spacings on the surface of Pd. Even for superstoichiometric D:Pd ratios no evidence for D-D spacings closer than that for the D_2 molecule (about 0.7 Å) was found under the most favorable model conditions, and in all of these studies no evidence was found to predict a sufficiently close spacing as required by the proposed rates for cold fusion.

C. Power Balance Calculations—The assertion by Profs. Fleischmann and Pons of excess energy production is derived from a power balance calculation based ultimately on Newton's Law of Cooling which may not be directly applicable in their situation. They use a submerged resistor to heat the electrolyte solution and assume a constant heat transfer coefficient which in turn is used to calculate the energy loss rate, $k_T \Delta T$, to the surrounding environment. However, total heat loss (especially from evacuated Dewar cells) may be dominated by evaporation of electrolyte which increases exponentially with temperature. Calculations under the assumption of evaporation-dominated heat loss suggest that there is, in actuality, little or no excess energy produced.

V. SUMMARY

In summary we have investigated electrochemical loading, pressure and temperature cycling, and the direct formation of superstoichiometric metal hydrides by implantation for the production of cold fusion. We have also probed in real time the kinetics of hydrogen isotope loading of electrochemical cells. In selected cases, electrochemical and gas cycling studies were carried out with neutron detection at extremely low background rates in underground facilities; unexpected spurious continuous and burst signals were observed in helium-3 detectors and it was found that multiple detectors provide a method to exclude such spurious signals which might otherwise be interpreted as evidence for cold fusion. We find no evidence to support the existence of cold fusion in these studies or to suggest the appropriateness of initiating an extensive program of research in this area.



John R. Huizenga
Co-Chairman, ERAB
Panel on Cold Fusion
1000 Independence Avenue S.W.
Washington DC 20585

September 10, 1989

Dear Dr. Huizenga:

Thank you for your letter of August 9. We are still much concerned by the very nature of this biased committee, and find it difficult to understand how any sensible selection procedure could come up with a list dominated by members who had already publicly declared (and viciously so) their verdict and opposition to this research. Nevertheless, we have on several occasions tried to comply with its requests since it determines the availability of badly-needed research funds for others working in this important area. You can therefore imagine our irritation with the subsequent statements made by members of your committee regarding the accuracy of our calorimetric measurements. We established the maximum error limits for our experiments before we submitted our preliminary publication, and we did indeed give these in the paper. In our more recent work, we have further established that we in fact somewhat overestimated the magnitude of the errors. These latest results will be published shortly, and we strongly suggest that you wait until this paper is available to the general scientific community before making ill-considered statements regarding the most important signature of the processes. Failure to do so will inevitably do further severe damage to the general public's opinion of science in general and the assessment procedures in particular in this country. As it has become quite clear that the committee cannot guarantee confidentiality of data which is going to be part of a publication, we will under no circumstances release this information before the paper has been reviewed.

With regards to your requests for information on our tritium results: nearly all of this information is in fact in our preliminary paper, to wit the size of the electrode, the total current, the description of the electrolytic cell, the total number of deuterons in the cell, the sample schedule and schedule of additions. The only information which you have requested that is not in the paper is the source of the D_2O used and its specific activity. These were, respectively, Cambridge Isotopes and 43 DPM/mL. We requested that the latter information be incorporated in our later corrections to the paper, but unfortunately, this was omitted by

Department of Chemistry

Henry Eyring Building
Salt Lake City, Utah 84112

the publishers. Professor Fleischmann has, however, sent several hundred corrected copies containing this information to laboratories around the world.

May we point out that the increase in activity in the solution, approximately 100DPM/mL, as given in the paper, is beyond the realms of possibility of any concentration factor due to electrolysis. Thermal balances on comparable cells showed excess enthalpy generations of 1 watt/cm³.

There are no known sources of tritium in the Henry Eyring South Chemistry Building. Furthermore our many blank experiments showed absolutely no trace of contamination.

We attempted to measure neutron production rates on cells identical to those used for generating tritium without success using our very limited equipment. The neutron count rates which we reported were obtained from a 0.4x10cm Pd electrode operated at 64mA/cm² which was giving excess heat somewhat above 1 watt/cm³. It should be noted that the volume of this electrode was 16 times that of the electrode used for the tritium measurements, and we presume that it is this that allowed statistically significant measurements for this electrode. The neutron counter was repeatedly moved between the cell location to adjacent locations where the background was taken, and, as we have stated, the count rate was 3 times the measured background. Counting was carried out for a total of a hundred hours in each location. In subsequent lectures, both Professor Fleischmann and I have pointed out that the excess neutrons generated in the cell arrive in bursts. We want to make the following additional observations: (1) attempts to detect neutrons will certainly fail if the electrodes do not give out excess heat (we believe that nearly all reported investigations were bound to fail on this score alone). (2) We fully accept that neutron measurements are very difficult and prone to error, however (3) attempts to impose complex verification procedures will fail unless they take into account that the neutrons arrive in bursts. That is, the verification procedure itself must have the time resolution of a typical BF₃ counter. We would recommend that work on this matter be carried out by those expert in this field when they can be supplied with cells which reliably give high levels of excess enthalpy generation. The present piecemeal research efforts in this area will lead not lead anywhere.

As you see, we are very willing to give information about matters which pertain to our published work.

With regard to Mr. Garwin's requests for data about the gamma ray spectra, we have nothing to add at this stage. Clearly we had severe problems with our gamma ray spectrometer presumably because of pile-up due to high energy gamma radiation. We are currently carrying out a lengthy investigation using an intrinsic Ge detector. We believe that this conclusively shows the generation of gamma rays due to thermalized neutrons generated by our electrodes and we will submit these results for publication in due course. Once again we must say we will not release this information to the committee before any such paper has been fully refereed.

Sincerely yours,


Stanley Pons
Professor

SP/sdp



Westinghouse
Savannah River Company

P.O. Box 616
Aiken, SC 29802

SRL-ELC-890097

August 31, 1989

Dr. William Woodard
Secretary, Cold Fusion Panel
ER-6, 3F-043
US Department of Energy
1000 Independence Avenue, SW
Washington, DC 20585

Dear Dr. Woodard:

The following information is supplied in response to letters of July 12 and August 9, 1989 from Dr. Huizenga.

- 1) D₂O comes from a stock of low activity moderator kept in our reactor moderator facilities. Tritium activity is 120 +/- 20 d/m/ml.
- 2) Our first cell which has operated for 3 months is a parallel
- &3) plate array with one 1" x 2" x .015" thick plate of palladium supported between two similar plates of platinum. The cell is contained in glass with Teflon™ and Pyrex™ supports. Electrolyte is 0.1 molar LiOD. Argon purge gas of 99.99% purity and 1 cc/sec flow rate carries decomposition gasses and vapor products from the cell. A cold trap and silica gel bed dry the gas which then passes across a mass spectrometer sampler. The gas is then mixed with a mixture of 90% argon plus 10% methane and flows through a 350cc proportional counter for tritium measurement. The cell is contained in a calorimeter which continuously monitors power within +/- 0.05 watts for a typical cell power of 6.5 watts and a current density of 0.040 amperes per cm². Total mass and energy into and out of the cell are carefully measured. Deuterium loaded into the pladium is computed from the mass spectrometer data.

Neutron flux is measured at the cell by two moderated helium -3 counters with a similar background counter located 6 meters away. Gamma flux is measured by a high resolution intrinsic germanium detector and a 3" diameter x 3" long sodium iodide scintillation detector at the cell. A 4" x 4" x 12" Polyscin detector monitors gamma background 6 meters from the cell.

- 4) D₂O is added every two days through the argon purge inlet.

5) Tritium Analysis

- a) No purification is done on samples. Most of the samples are condensed D₂O vapor.
 - b) Prior to assembly, all cell components are checked for tritium contamination with a windowless gas proportional counter.
 - c) Liquid tritium counting is done by adding sample water to scintillation cocktail and counting in a scintillation counter. Liquid is counted once a week or when a significant change is observed in the cell.
 - d) Tritium content of electrolysis product gas is continuously monitored by a flow-through proportional counter.
 - e) No tritium has been observed above expected levels due to electrolytic concentration.
- 6) No significant neutron signals have been observed above background.
- 7) Total energy input has been balanced by total energy output within +/- 2%.
- 8) No tritium sources are located in the building where the cells are operated.

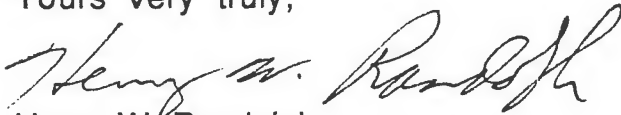
Electrical characteristics of the cell have not been stable and have been severely affected by addition of small amounts of poisons. Workers finding tritium have reported that only a small percentage of cells produce tritium after a long period of time. Our single cell is, therefore, not a statistically adequate sample for the observed effects. We are assembling 12 cells with 1 cm² palladium cathodes and nickel anodes. We have obtained components for another cell from Dr. John Bockris at Texas A&M and have started this cell into operation. Forty-five additional cells are being built using pure components to obtain careful control of a variety of poisons. No argon purge will be used with these cells and heat will not be measured. Some cells will be made with catalytic deuterium-oxygen recombiners. Also under construction is equipment to monitor tritium content of off-gas products from these cells.

Dr. William Woodard
Page 3
August 31, 1989

We are conducting an optical microscope study of paladium surfaces during electrolysis of light water and we have found that formation of hydrogen and oxygen gas occurs at very small and widely separated locations on the surface. This may be significant to electrode behavior after poisoning by extremely small amounts of other elements.

Should you need additional information, please let me know.

Yours very truly,



Henry W. Randolph
Physicist
Sr. Research Associate
Westinghouse Savannah River Laboratory

HWR:jfc

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BROOKHAVEN NATIONAL LABORATORY

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Upton, Long Island, New York 11973

(516) 282-
FTS 666-2772

Office of the Director

September 18, 1989

Dr. John R. Huizenga
Co-Chairman, Cold Fusion Panel
Energy Research Advisory Board
1000 Independence Avenue, S.W.
Washington, D.C. 20585

Dear John:

Enclosed you will find the material you requested summarizing our past and present research on cold fusion.

You will shortly see that I have written an article with Bob Crease for the Sunday Times magazine section on cold fusion. Hope you enjoy it.

Sincerely,



N. P. Samios
Director

Enclosure

New Energy Times Archive

MATERIALS USED IN COLD FUSION EXPERIMENTS

PRINCIPAL INVESTIGATORS:

R. Davis, J. R. Johnson, J.J. Reilly and R. Stoenner.

ORGANIZATION:

BNL

SHORT DESCRIPTION OF EXPERIMENT:

Determination of tritium content of deuterium stored in metal deuteride reservoirs and deuterium gas cylinders.

RESULTS AND COMMENTS:

For a number of years we have stored D_2 for laboratory use, as a metal deuteride. The tritium content of such D_2 was found to be substantially higher than that from the gas cylinders known to have been used to supply most of the stored deuterium. However, it is not certain that they were the only cylinders which were used. While the cylinder dated 1/10/78 (see table) was not used to charge the reservoirs it indicates that some D_2 cylinders may have a substantial T content. Further, it is also possible that some T contamination by other means occurred over the course of years.

TRITIUM CONTENT OF METAL DEUTERIDE RESERVOIRS

Reservoir	weight, g	T. content ct (day ⁻¹ ml ⁻¹)	Reservoir Age, y
FeTiD _x	1500	966+/-14	11
FeTiD _x	1500	859+/-13	12
REMi ₅ D _x ⁺		1226+/-14	>5
Fe _{.7} TiMn _{.2}		82+/-4	>5

+ RE = Rare earth components derived from misch metal.

TRITIUM CONTENT OF D₂ GAS CYLINDERS

Cylinder #	T content ct (day ⁻¹ ml ⁻¹)
545473*	75+/-3
SG2564*	70+/-3
545462*	82+/-
g-16055	145+/-5
1/10/78	2051+/-19

*Known to have been used to charge above reservoirs.

MATERIALS USED IN COLD FUSION EXPERIMENTS

PRINCIPAL INVESTIGATOR: Stephen W. Feldberg

ORGANIZATION: BNL (DAS)

SHORT DESCRIPTION OF TYPE OF EXPERIMENT:

Search for neutrons and/or gammas ($2 \rightarrow 30$ MeV) associated with electrolysis of D_2O (+ $0.25M LiOD$) at Pd electrode: (Total current $\sim 2A$; current density $\sim 0.6 A/cm^2$)
Also a crude estimate of power balance.
Pd was annealed and pre-deuterated to $\sim 0.7 H/Pd$

RESULTS AND COMMENTS:

No neutrons; no gammas; no heat ($\pm 10\%$)

Cell ran for ~ 2 weeks; study was carried out during 2nd week. Since Pd electrode was a large (5.8 gm) cylinder (0.6 cm d x 1.5 cm) it might be argued that we did not electrolyse long enough. Predeuteration should. Cell had a Fuel-cell ($H_2 + O_2$) catalyst so total loss of D_2O after 2 weeks at 2 Amps was $< 1ml$ (out of $25 ml$).

I. MATERIALS USED FOR ELECTROCHEMICAL EXPERIMENTS (please complete one sheet for each experiment)

	CATHODES	ANODES (Corresponding)
MATERIAL	Pd	Pt
PURITY	99.9+	99.9+
ALLOYING ELEMENTS	-	-
SOURCE OF MATERIAL	Stock	Stock
PREPARATION CAST OR WROUGHT ANNEALED ATMOSPHERE VACUUM SPECIAL TREATMENT	Outgassed at 200° c/10 ⁻⁶ Torr Predeuterated at 300° and 20 Atm D ₂ and cooled to ~approximately room temperature Final comp (by weight; D/Pd ~0.7)	
CHARACTERIZATION STRUCTURAL CHEMICAL BEFORE OR AFTER USE METHODS RESULTS		
NOTABLE OBSERVATIONS		
D / METAL RATIO ATTAINED		
EXPERIMENT YIELDED ^{Excess} HEAT	_____yes	____X____no
NEUTRONS	_____yes	____X____no
TRITIUM	_____yes	_____no
HELIUM	_____yes	_____no
GAMMAS		____X____no

MATERIALS USED IN COLD FUSION

PRINCIPAL INVESTIGATORS

S.W. Feldberg, S.D. Hoblit, J. R. Johnson, O.C. Kistner,
G. Matone, L. Miceli, J.J. Reilly, A.M. Sandorfi, C.E. Thorn,
C.S. Whisnant

ORGANIZATION

BNL

SHORT DESCRIPTION OF EXPERIMENT

Search for emission of neutrons in metal/deuterium systems using a NaI detector with an efficiency of about 1 %. The experiments were carried under varying conditions of pressure, temperature and solid compositions.

RESULTS AND COMMENTS

No counts above background which could be attributed to a D-D fusion event were observed. However, it should be noted that the counting arrangement required that the counts be integrated over a five minute period; thus detection of random neutron bursts of less than 2000 would be unlikely because of the low efficiency and a background of 0.5 ± 0.1 neutrons/sec.

RECEIVED

AUG 30 1989

DEPT. OF PHYSICS

SUMMARY
METAL/DEUTERIUM SYSTEMS

EXP#	CPD.	WT. g	P, D ₂ atm.	T, K	REMARKS
2322	VD ₂	45.9	34	77-293	
2322	VD _X ⁺	45.9	1-16	293-358	x=2-.8
2323	TiD ₂	70.2	<<1	77-293	
2324	Ti	70	20	77-293	
2324	TiD _{.1} ⁺	70	20->>1	293-575	reacted
2324	TiD _{.2} ⁺	70	20->>1	293-601	reacted
2329	Ti*	48	10	77	
2326	LaD ₃	25	32-1	298-725	
	FeTiD-1	1000	15	293	
	FeTiD-2	1000	15	293	

+ Reacted in situ.

* Commercial grade, 90% Ti, 6%Al, 4% V

II. MATERIALS USED FOR GASEOUS CHARGING EXPERIMENTS (please complete one sheet for each experiment)

MATERIAL \checkmark D_2 (46g) Expt # 2322

PURITY 99.7% \checkmark

ALLOYING
ELEMENTS

SOURCE OF
MATERIAL Union Carbide Corp

PREPARATION

CAST OR WROUGHT

ANNEALED

ATMOSPHERE

VACUUM Outgassed @ 450°C

SPECIAL TREATMENT

CHARACTERIZATION

STRUCTURAL

CHEMICAL \checkmark

BEFORE OR AFTER USE Before
METHODS Measure D_2 uptake.

RESULTS

No counts above background.

NOTABLE
OBSERVATIONS

D / METAL RATIO ATTAINED

EXPERIMENT YIELDED HEAT

NEUTRONS

TRITIUM

HELIUM

_____yes

_____yes

_____yes

_____yes

_____ \checkmark no

_____ \checkmark no

_____ \checkmark no

_____ \checkmark no

II. MATERIALS USED FOR GASEOUS CHARGING EXPERIMENTS (please complete one sheet for each experiment)

MATERIAL VD_x (46g)

PURITY 99.7% V

ALLOYING
ELEMENTS

SOURCE OF
MATERIAL Union Carbide Corp.

PREPARATION
CAST OR WROUGHT
ANNEALED
ATMOSPHERE
VACUUM
SPECIAL TREATMENT

Outgassed @ 450°C

CHARACTERIZATION
STRUCTURAL
CHEMICAL ✓

BEFORE OR AFTER USE Before + during experiment
METHODS Measure D_2 absorption + evolution.

RESULTS

Decomposed in situ according to
the following reaction



No counts above background.

NOTABLE
OBSERVATIONS

D/METAL RATIO ATTAINED

EXPERIMENT YIELDED HEAT
NEUTRONS
TRITIUM
HELIUM

_____yes	_____no
_____yes	_____no
_____yes	_____no
_____yes	_____no

II. MATERIALS USED FOR GASEOUS CHARGING EXPERIMENTS (please complete one sheet for each experiment)

MATERIAL $T_1 D_2$ (70g)

PURITY 99.9%

ALLOYING
ELEMENTS

SOURCE OF
MATERIAL Metron Inc.

PREPARATION
CAST OR WROUGHT
ANNEALED
ATMOSPHERE
VACUUM
SPECIAL TREATMENT

CHARACTERIZATION
STRUCTURAL

CHEMICAL ☒

BEFORE OR AFTER USE Before

METHODS Measure D_2 uptake.

RESULTS

No counts above background.

NOTABLE
OBSERVATIONS

D/METAL RATIO ATTAINED

EXPERIMENT YIELDED HEAT
NEUTRONS
TRITIUM
HELIUM

_____yes
_____yes
_____yes
_____yes

_____☒no
_____☒no
_____☒no
_____☒no

II. MATERIALS USED FOR GASEOUS CHARGING EXPERIMENTS (please complete one sheet for each experiment)

MATERIAL $TiD_{0.1}$ (70g)

PURITY 99.9% Ti

ALLOYING
ELEMENTS

SOURCE OF
MATERIAL Metron Inc.

PREPARATION
CAST OR WROUGHT
ANNEALED
ATMOSPHERE
VACUUM
SPECIAL TREATMENT

CHARACTERIZATION
STRUCTURAL
CHEMICAL ✓
BEFORE OR AFTER USE
METHODS
RESULTS

Reacted in situ via
 $Ti + .05D_2 \rightarrow TiD_{0.1}$

NOTABLE
OBSERVATIONS

D/METAL RATIO ATTAINED

EXPERIMENT YIELDED HEAT	Chemical ✓	yes	no
NEUTRONS		yes	✓ no
TRITIUM		yes	✓ no
HELIUM		yes	✓ no

II. MATERIALS USED FOR GASEOUS CHARGING EXPERIMENTS (please complete one sheet for each experiment)

MATERIAL $TiD_{0.2}$ (70g)

PURITY 99.9% Ti

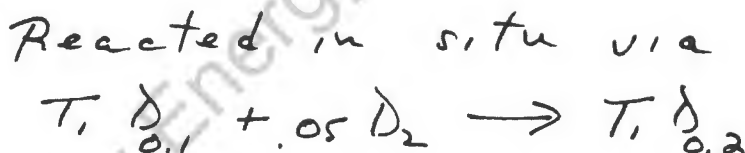
ALLOYING
ELEMENTS

SOURCE OF
MATERIAL

PREPARATION
CAST OR WROUGHT
ANNEALED
ATMOSPHERE
VACUUM
SPECIAL TREATMENT

CHARACTERIZATION
STRUCTURAL
CHEMICAL ✓
BEFORE OR AFTER USE
METHODS
RESULTS

During use



NOTABLE
OBSERVATIONS

D/METAL RATIO ATTAINED

EXPERIMENT YIELDED HEAT	Chemical ✓	yes	no
NEUTRONS		yes	✓ no
TRITIUM		yes	✓ no
HELIUM		yes	✓ no

II. MATERIALS USED FOR GASEOUS CHARGING EXPERIMENT: (please complete one sheet for each experiment)

MATERIAL $T_1(48g) + D_2 @ 10 atm (298 K)$

PURITY 90 %

ALLOYING ELEMENTS 6 % Al, 4 % V

SOURCE OF MATERIAL

PREPARATION

CAST OR WROUGHT

ANNEALED

ATMOSPHERE

VACUUM

SPECIAL TREATMENT

CHARACTERIZATION

STRUCTURAL

CHEMICAL

BEFORE OR AFTER USE

METHODS

RESULTS

No counts above background.

NOTABLE OBSERVATIONS

D / METAL RATIO ATTAINED

EXPERIMENT YIELDED HEAT

NEUTRONS

TRITIUM

HELIUM

_____yes

_____yes

_____yes

_____yes

_____☒no

_____☒no

_____☒no

_____☒no

II. MATERIALS USED FOR GASEOUS CHARGING EXPERIMENTS (please complete one sheet for each experiment)

MATERIAL La D_3 (25g)

PURITY La , 99 %

ALLOYING
ELEMENTS

SOURCE OF
MATERIAL *United Mineral & Chemical Corp.*

PREPARATION
CAST OR WROUGHT
ANNEALED
ATMOSPHERE
VACUUM
SPECIAL TREATMENT

CHARACTERIZATION
STRUCTURAL
CHEMICAL ✓

BEFORE OR AFTER USE *Before*
METHODS *Measured D_2 uptake*
RESULTS

No counts above background.

NOTABLE
OBSERVATIONS

D/METAL RATIO ATTAINED

EXPERIMENT YIELDED HEAT
NEUTRONS
TRITIUM
HELIUM

_____yes
_____yes
_____yes
_____yes

_____✓no
_____✓no
_____✓no
_____✓no

II. MATERIALS USED FOR GASEOUS CHARGING EXPERIMENTS (please complete one sheet for each experiment)

MATERIAL $FeTiD_{0.2}$

PURITY 99% $FeTi$

ALLOYING
ELEMENTS

SOURCE OF
MATERIAL NL Industries

PREPARATION
CAST OR WROUGHT cast
ANNEALED
ATMOSPHERE
VACUUM
SPECIAL TREATMENT

CHARACTERIZATION
STRUCTURAL
CHEMICAL ✓

BEFORE OR AFTER USE

METHODS D content estimated from pressure/temperature
RESULTS correlation.

No counts above background

NOTABLE
OBSERVATIONS

D/METAL RATIO ATTAINED

EXPERIMENT YIELDED HEAT
NEUTRONS
TRITIUM
HELIUM

_____yes
_____yes
_____yes
_____yes

_____✓no
_____✓no
_____✓no
_____✓no

II. MATERIALS USED FOR GASEOUS CHARGING EXPERIMENTS (please complete one sheet for each experiment)

MATERIAL $FeTiD_{21}$ (1000g)

PURITY ~ 99% $FeTi$.

ALLOYING
ELEMENTS

SOURCE OF
MATERIAL NL Industries

PREPARATION
CAST OR WROUGHT
ANNEALED
ATMOSPHERE
VACUUM
SPECIAL TREATMENT

CHARACTERIZATION
STRUCTURAL
CHEMICAL ✓

BEFORE OR AFTER USE

METHODS From pressure/temperature correlation

RESULTS

No counts above background

NOTABLE
OBSERVATIONS

D/METAL RATIO ATTAINED

EXPERIMENT YIELDED HEAT
NEUTRONS
TRITIUM
HELIUM

_____yes

_____yes

_____yes

_____yes

_____☒_____no

_____☒_____no

_____☒_____no

_____☒_____no

MATERIALS USED IN COLD FUSION EXPERIMENTS

PRINCIPAL INVESTIGATOR:

M. Gai^{*}, S. L. Rugari^{*}, R. H. France^{*}, B. J. Lund^{*}, Z. Zhao^{*}, A. J. Davenport
H. S. Isaacs[†] & K. G. Lynn[‡]

ORGANIZATION:

^{*} A. W. Wright Nuclear Structure Laboratory, Yale University, New Haven, Connecticut 06511, USA

[†] Department of Applied Science, Brookhaven National Laboratory, Upton, New York 11973, USA

[‡] Department of Physics and Applied Science, Brookhaven National Laboratory Upton, New York 11973, USA

SHORT DESCRIPTION OF TYPE OF EXPERIMENT:

Here we report on a search for neutrons and γ -rays emitted during conjectured fusion of $d+d$ and $p+d$ in various metals into which deuterium has been incorporated ('deuterided' metals). Measurements were carried out with an array of low-background, high-sensitivity neutron detectors and two large high-efficiency NaI(Tl) γ -ray detectors.

RESULTS AND COMMENTS:

Neutron and γ -ray emission from a variety of electrochemical cells (running continuously for up to two weeks) have been measured using a sensitive detection system with a very low background. Titanium alloy powder deuterided at room temperature and high pressure was also used for comparison. No statistically significant deviation from the background was observed in either γ -ray or neutron detectors. The estimated neutron flux in this experiment is at least a factor of 50 times smaller than that reported by Jones *et al.* and about one million times smaller than that reported by Fleischmann *et al.* The results suggest that a significant fraction of the observed neutron events are associated with cosmic rays.

I. MATERIALS USED FOR ELECTROCHEMICAL EXPERIMENTS (please complete one sheet for each experiment)

	CATHODES	ANODES (Corresponding)
MATERIAL	Pd	Pt
PURITY	>99.9	
ALLOYING ELEMENTS	—	

SOURCE OF MATERIAL	BNL stock
-----------------------	-----------

PREPARATION	
CAST OR WROUGHT	cold worked
ANNEALED	
ATMOSPHERE	D ₂
VACUUM	300°C / 120 psi
SPECIAL TREATMENT	

CHARACTERIZATION	
STRUCTURAL	
CHEMICAL	NONE
BEFORE OR AFTER USE	
METHODS	
RESULTS	

NOTABLE OBSERVATIONS	NONE
-------------------------	------

D / METAL RATIO ATTAINED

EXPERIMENT YIELDED HEAT	
NEUTRONS	
TRITIUM	
HELIUM	

<input checked="" type="checkbox"/> yes	<input type="checkbox"/> no
<input type="checkbox"/> yes	<input checked="" type="checkbox"/> no
<input type="checkbox"/> yes	<input checked="" type="checkbox"/> no
<input type="checkbox"/> yes	<input checked="" type="checkbox"/> no

But not studied. This does not mean excess heat

I. MATERIALS USED FOR ELECTROCHEMICAL EXPERIMENTS (please complete one sheet for each experiment)

CATHODES

ANODES
(Corresponding)

MATERIAL

Pd

Pt

PURITY

>99.9

ALLOYING
ELEMENTS

—

SOURCE OF
MATERIAL

BNL stock

PREPARATION

CAST OR WROUGHT

~~ANNEALED~~

ATMOSPHERE

~~VACUUM~~

SPECIAL TREATMENT

ANNEALED AT 900°C

IN VACUUM

pre anodized after etching

CHARACTERIZATION

STRUCTURAL

CHEMICAL

BEFORE OR AFTER USE

METHODS

RESULTS

NONE

NOTABLE
OBSERVATIONS

NONE

D / METAL RATIO ATTAINED

EXPERIMENT YIELDED HEAT

NEUTRONS

TRITIUM

HELIUM

☒ yes

----- yes

----- yes

----- yes

----- no

☒ no

☒ no

☒ no

But not studied. This does not mean excess heat

I. MATERIALS USED FOR ELECTROCHEMICAL EXPERIMENTS (please complete one sheet for each experiment)

	CATHODES	ANODES (Corresponding)
MATERIAL	Pd	Pt
PURITY	>99.9	
ALLOYING ELEMENTS	—	

SOURCE OF MATERIAL	BNL stock
-----------------------	-----------

PREPARATION	
CAST OR WROUGHT	
ANNEALED	ANNEALED 1000°C
ATMOSPHERE	ARGON
VACUUM	
SPECIAL TREATMENT	preanodized after etching

CHARACTERIZATION
STRUCTURAL
CHEMICAL
BEFORE OR AFTER USE
METHODS
RESULTS

NOTABLE
OBSERVATIONS

NONE

D / METAL RATIO ATTAINED

EXPERIMENT YIELDED HEAT
NEUTRONS
TRITIUM
HELIUM

<input checked="" type="checkbox"/> yes	<input type="checkbox"/> no
<input type="checkbox"/> yes	<input checked="" type="checkbox"/> no
<input type="checkbox"/> yes	<input checked="" type="checkbox"/> no
<input type="checkbox"/> yes	<input checked="" type="checkbox"/> no

But not
studied. This does
not mean excess heat

I. MATERIALS USED FOR ELECTROCHEMICAL EXPERIMENTS (please complete one sheet for each experiment)

	CATHODES	ANODES (Corresponding)
MATERIAL	Ti	Pt
PURITY	>99.9	
ALLOYING ELEMENTS	—	

SOURCE OF MATERIAL	BNL stock
-----------------------	-----------

PREPARATION	
CAST OR WROUGHT	cold rolled.
ANNEALED	
ATMOSPHERE	
VACUUM	
SPECIAL TREATMENT	

CHARACTERIZATION	
STRUCTURAL	
CHEMICAL	
BEFORE OR AFTER USE	pre etched.
METHODS	
RESULTS	

NOTABLE OBSERVATIONS	NONE
-------------------------	------

D / METAL RATIO ATTAINED

EXPERIMENT YIELDED HEAT	
NEUTRONS	
TRITIUM	
HELIUM	

_____x_____yes	_____no
_____yes	_____x_____no
_____yes	_____x_____no
_____yes	_____✓_____no

But not studied. This does not mean excess heat

MATERIALS USED IN COLD FUSION

PRINCIPAL INVESTIGATORS

J. R. Johnson, K. Lynn, J.J. Reilly, K. Ritley and M. Weber

ORGANIZATION

BNL

SHORT DESCRIPTION OF EXPERIMENT

Search for emission of neutron bursts in metal/deuterium systems of various solid compositions under varying regimes of pressure and temperature. A burst count is defined as the number counts which occur within a period of 64 μ s after an initial count. A shielded BF₃ counter was used with a background of ≈ 1 count/s.

RESULTS AND COMMENTS

Several counting bursts were observed in all experiments including blanks. It was determined that a more sophisticated counting arrangement and electronic signal processing will be required before any certain conclusions can be made regarding the rate of D-D fusion in these systems

SUMMARY
METAL/DEUTERIUM SYSTEMS

EXP.#	CPD.	WT. g atm.	P, D ₂	T, K	Burst* max.	REMARKS
1	VD ₂	45.9	34	133-295	16	Decomp.
2	Ti ⁺	61	15	77-290	3	
3	TiD _{0.1}	48	10	77-298	7	
4	YD ₃	20	15	77-290	7	
5	FeTiMnH _x	100	15	158-295	24	Blank
6	YD ₃	20	15			
	VD ₂	45.9	34	135-295	5	3 Reactors
	LaD ₃	25	15			
7!	TiCr _{1.8}	12				
	TiMn _{1.5}	4				
	Ti _{1.1} Cr _{0.9} Mn	4				
	ZrCo ₂	2	0-10	190-314	3	Fract Fusion Single Reactor
	ZrCr ₂	5				
8!	TiMn _{1.4}	4				
	TiCr _{1.8}	17				
	TiCr ₂	7	0-15	223-317	167	Fract Fusion Single Reactor
	Ti _{1.1} Cr _{0.9} Mn	9				

*In 50 or 100 second counting period

+Commercial Alloy Ti-6%Al-4%V

!These materials fracture upon reacting with D₂.

1. MATERIALS USED FOR GASEOUS CHARGING EXPERIMENTS (please complete one sheet for each experiment)

MATERIAL VD_2 (46 g)

PURITY 99.7 % V

ALLOYING
ELEMENTS

SOURCE OF MATERIAL Union Carbide Corp.

PREPARATION

CAST OR WROUGHT

ANNEALED

ATMOSPHERE

VACUUM

Outgassed @ 450°C

SPECIAL TREATMENT

CHARACTERIZATION

STRUCTURAL

CHEMICAL

BEFORE OR AFTER USE

Before

METHODS

measure $\Delta\alpha$ uptake.

RESULTS

Max. burst count = 16

NOTABLE

OBSERVATIONS

Sample was decomposed in situ according to following reaction



1/METAL RATIO ATTAINED

EXPERIMENT YIELDED HEAT*

NEUTRONS

_____ yes

_____ ☒ no

TRITIUM

2. _____ yes

_____ no

HELIUM

2. _____ yes

_____ no

_____ yes

_____ no

Heat was absorbed during decomposition.

II. MATERIALS USED FOR GASEOUS CHARGING EXPERIMENTS (please complete one sheet for each experiment)

MATERIAL $TiD_{0.1}$ (48g) + D_2 (150751a @ 298K)

PURITY 99.5%

ALLOYING
ELEMENTS

SOURCE OF
MATERIAL Metron Inc.

PREPARATION
CAST OR WROUGHT
ANNEALED
ATMOSPHERE
VACUUM
SPECIAL TREATMENT

CHARACTERIZATION
STRUCTURAL
CHEMICAL
BEFORE OR AFTER USE
METHODS
RESULTS

NOTABLE
OBSERVATIONS

D/METAL RATIO ATTAINED

EXPERIMENT YIELDED HEAT
NEUTRONS
TRITIUM
HELIUM

_____ yes
_____ ? yes
_____ ? yes
_____ ? yes

_____ ✓ no
_____ no
_____ no
_____ no

II. MATERIALS USED FOR GASEOUS CHARGING EXPERIMENTS (please complete one sheet for each experiment)

MATERIAL T_1 (61g) + D_2 (225 psia)

PURITY 90%

ALLOYING ELEMENTS 6% Al, 4% V

SOURCE OF MATERIAL

PREPARATION Shavings

CAST OR WROUGHT

ANNEALED

ATMOSPHERE

VACUUM

SPECIAL TREATMENT

Outgassed @ 240°C

CHARACTERIZATION

STRUCTURAL

CHEMICAL

BEFORE OR AFTER USE

METHODS

RESULTS

Max. burst count = 3

NOTABLE OBSERVATIONS

D/METAL RATIO ATTAINED

EXPERIMENT YIELDED HEAT

NEUTRONS

TRITIUM

HELIUM

yes

2. yes

2. yes

2. yes

✓ no

no

no

no

1. MATERIALS USED FOR GASEOUS CHARGING EXPERIMENTS (please complete one sheet for each experiment)

MATERIAL 20g Y D₃

PURITY 99+% Y

ALLOYING
ELEMENTS

SOURCE OF
MATERIAL

Johnson Matthey

PREPARATION
CAST OR WROUGHT
ANNEALED
ATMOSPHERE
VACUUM
SPECIAL TREATMENT

Direct reaction of D₂ with Y

CHARACTERIZATION

STRUCTURAL

CHEMICAL ✓

BEFORE OR AFTER USE

Before

METHODS

D₂ uptake

RESULTS

Max. burst count = 8

NOTABLE

OBSERVATIONS

/ METAL RATIO ATTAINED

PERIMENT YIELDED HEAT

NEUTRONS

TRITIUM

HELIUM

____ yes

____ 2 yes

____ 2 yes

____ 2 yes

____ ✓ no

____ no

____ no

____ no

II: MATERIALS USED FOR GASEOUS CHARGING EXPERIMENTS (please complete one sheet for each experiment)

MATERIAL FeTi (100g) + H

PURITY 98 % FeTi

ALLOYING ELEMENTS 1 % Mn

SOURCE OF MATERIAL Cannon Muskegon Corp

PREPARATION

CAST OR WROUGHT Cast

ANNEALED

ATMOSPHERE

VACUUM

SPECIAL TREATMENT

CHARACTERIZATION

STRUCTURAL ☒ x-Ray diffraction

CHEMICAL ☒ by wet analysis

BEFORE OR AFTER USE

METHODS

RESULTS

max burst count = 17

NOTABLE OBSERVATIONS

Blank experiment

D/METAL RATIO ATTAINED H/Ti ~~1.8~~ ≈ 1.8

EXPERIMENT YIELDED HEAT

NEUTRONS

TRITIUM

HELIUM

yes

yes

yes

yes

☒ no

no

no

no

11. MATERIALS USED FOR GASEOUS CHARGING EXPERIMENTS (please complete one sheet for each experiment)

MATERIAL 20 g YD_3 , 45.9 g VD_2 , 25g LaD_3

PURITY All metals 99+ %

ALLOYING
ELEMENTS

SOURCE OF
MATERIAL

Y - Johnson Mathey
V - Union Carbide Corp
La - United Mineral & Chemical Corp

PREPARATION

Direct reaction of metal with D_2

CAST OR WROUGHT

ANNEALED

ATMOSPHERE

VACUUM

SPECIAL TREATMENT

CHARACTERIZATION

STRUCTURAL

CHEMICAL

BEFORE OR AFTER USE

METHODS

RESULTS

Max. burst count = 5

NOTABLE

OBSERVATIONS

Samples were contained in separate reactor but counted simultaneously

1 / METAL RATIO ATTAINED

EXPERIMENT YIELDED HEAT

NEUTRONS

TRITIUM

HELIUM

_____ yes

2 _____ yes

2 _____ yes

2 _____ yes

✓ _____ no

_____ no

_____ no

_____ no

1. MATERIALS USED FOR GASEOUS CHARGING EXPERIMENTS (please complete one sheet or each experiment)

MATERIAL 12g $Ti_{0.8}$, 4g $Ti_{1.5}$, 4g $Ti_{1.1}$, 0.9 Mn, 2g ZrC
PURITY 5g ZrC₂, 4g $Ti_{1.4}$

ALLOYING
ELEMENTS

SOURCE OF MATERIAL BNL

PREPARATION From the elements by arc melting
CAST OR WROUGHT
ANNEALED ☒
ATMOSPHERE
VACUUM
SPECIAL TREATMENT

CHARACTERIZATION X-ray diffraction
STRUCTURAL ☒
CHEMICAL
BEFORE OR AFTER USE Before
METHODS
RESULTS

Samples were introduced as small buttons into a single reactor & outgassed @450°. Reactor was transferred to counter and D₂ introduced. Max burst count = 3

NOTABLE OBSERVATIONS Samples break apart doing ~~D~~ D absorption.

D/METAL RATIO ATTAINED

EXPERIMENT YIELDED HEAT	Chemical <input checked="" type="checkbox"/>	yes	no
NEUTRONS	2	yes	no
TRITIUM	2	yes	no
HELIUM	2	yes	no

1. MATERIALS USED FOR GASEOUS CHARGING EXPERIMENTS (please complete one sheet for each experiment)

MATERIAL 17g $TiC_{1.8}$, 7g TiC_2 , 9g $Ti_{1.1}C_{0.9}$ mm

PURITY

ALLOYING
ELEMENTS

SOURCE OF
MATERIAL

BNL

PREPARATION
CAST OR WROUGHT
ANNEALED
ATMOSPHERE
VACUUM
SPECIAL TREATMENT

From the elements by arc melting

CHARACTERIZATION

STRUCTURAL ☒

x-ray diffraction

CHEMICAL

BEFORE OR AFTER USE

METHODS

RESULTS

Introduced as buttons into single reactor.
Outgassed $\sim 400^\circ C$. Reactor was transferred
to counter and D_2 introduced. Samples cycled
through several absorption/desorption reactions.
Max. burst count = 167

NOTABLE

OBSERVATIONS

Largest burst count noted.

1/METAL RATIO ATTAINED

EXPERIMENT YIELDED HEAT Chemical

NEUTRONS

TRITIUM

HELIUM

☒ yes

-----no

☒ yes

-----no

☒ yes

-----no

☒ yes

-----no

MATERIALS USED IN COLD FUSION EXPERIMENTS

PRINCIPAL INVESTIGATOR: K. G. Lynn

ORGANIZATION: Brookhaven National Laboratory

SHORT DESCRIPTION OF TYPE OF EXPERIMENT: We monitor the electrolytic process in heavy water with a charged particle sensitive detector (2mm thick surface barrier detector). Any energetic electrons or protons emitted during possible fusion reactions are detected. A 5" X 5" NaI scintillator is utilized to detect high energy γ -rays.

RESULTS AND COMMENTS:

1) NaI scintillator:

long term (days) measurements while the electrolytic cell is operating show no distinguishable effect other than natural background of the environment. The region in the NaI detector around 20 to 30 MeV is essentially flat within statistical errors of 350 counts in $\approx 3.5 \times 10^5$ sec.

2) Surface barrier detector:

It was set up to detect energetic particles up to 1.2 MeV. When the cell was running the count rate in the region below ≈ 150 keV increased. This was attributed to the increase noise level of the detector due to the increase temperature of the cell. (The detector is immersed in the electrolyte). Further investigations of this effect are under way.

RECEIVED

SEP 6 1989

DEPT. OF PHYSICS

I. MATERIALS USED FOR ELECTROCHEMICAL EXPERIMENTS (please complete one sheet for each experiment)

	CATHODES	ANODES (Corresponding)	
MATERIAL	Pd foil	Pt	
PURITY	commercial	commercial	
ALLOYING ELEMENTS	none	none	
SOURCE OF MATERIAL			
PREPARATION			
CAST OR WROUGHT	foil $\approx 0.005"$	wire	
ANNEALED	700° C		
ATMOSPHERE	in Ar		
VACUUM			
SPECIAL TREATMENT			
CHARACTERIZATION			
STRUCTURAL			
CHEMICAL			
BEFORE OR AFTER USE		black coating on	
METHODS		surface is carbon	
RESULTS		no pre charging	
NOTABLE OBSERVATIONS			
D / METAL RATIO ATTAINED	not measured		
EXPERIMENT YIELDED HEAT	_____yes	_____no	
NEUTRONS	_____yes	_____no	no charged
TRITIUM	_____yes	_____no	particles were
HELIUM	_____yes	_____no	observed so far

II: MATERIALS USED FOR GASEOUS CHARGING EXPERIMENTS (please complete one sheet for each experiment)

MATERIAL

PURITY

ALLOYING
ELEMENTS

SOURCE OF
MATERIAL

PREPARATION

CAST OR WROUGHT

ANNEALED

ATMOSPHERE

VACUUM

SPECIAL TREATMENT

CHARACTERIZATION

STRUCTURAL

CHEMICAL

BEFORE OR AFTER USE

METHODS

RESULTS

NOTABLE
OBSERVATIONS

D/METAL RATIO ATTAINED

EXPERIMENT YIELDED HEAT

NEUTRONS

TRITIUM

HELIUM

-----yes

-----yes

-----yes

-----yes

---X---no

---X---not measured in this

---X---no equipment

-----no

MATERIALS USED IN COLD FUSION EXPERIMENTS

PRINCIPAL INVESTIGATOR: Kelvin G. Lynn

ORGANIZATION: Brookhaven National Laboratory, Upton, New York 11973

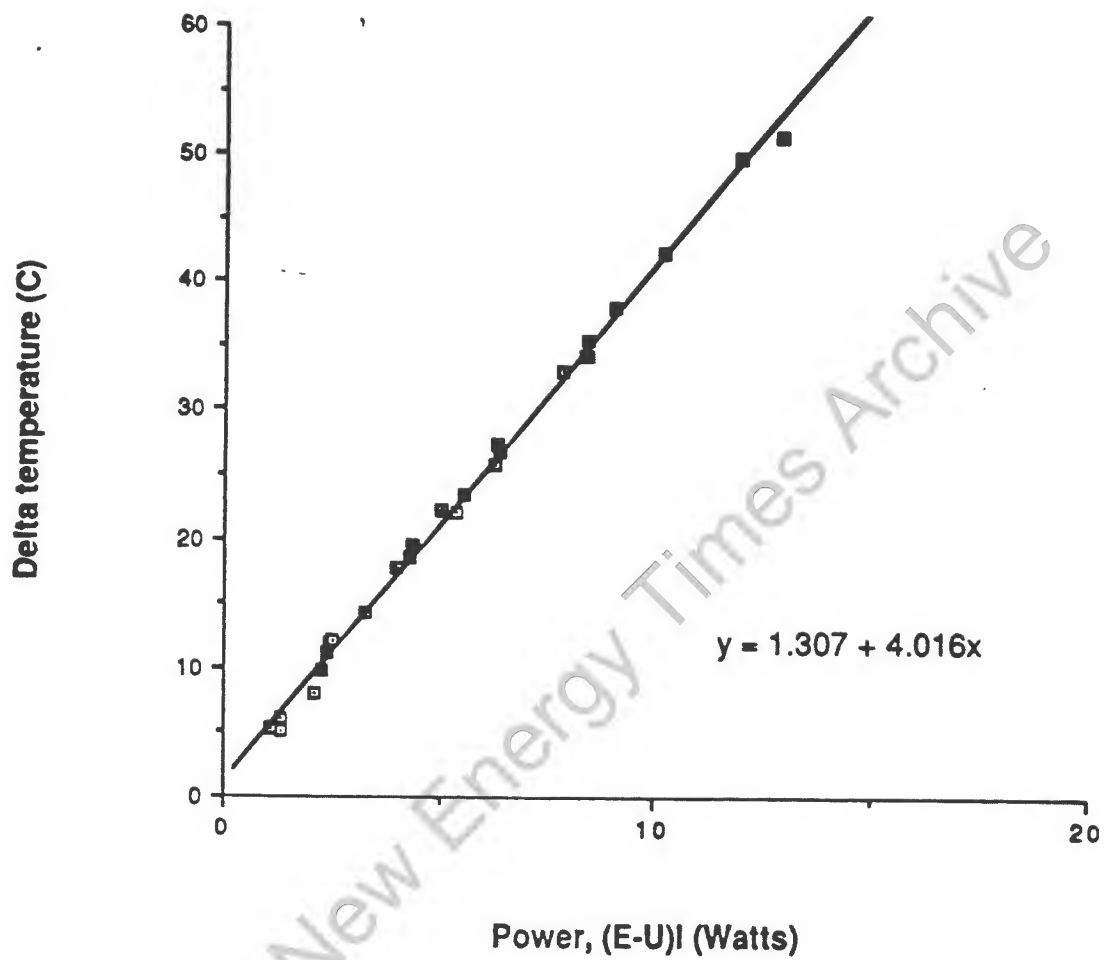
SHORT DESCRIPTION OF TYPE OF EXPERIMENT:

Calorimetry Measurements - 8 - D₂O cells - continuous monitoring
Mounted with thermal probes
Sensitivity ~ 10-20 mWatts with approximately 10-30 Watts into cells

RESULTS AND COMMENTS:

All cells have high surface area Pt recombiners to eliminate D₂O loss by electrolysis; and avoid Tritium concentration. All these cells are mounted in a temperature controlled reservoir ($\pm 1^\circ\text{C}$). Tritium measurements are made weekly. The electrodes are various diameters and metallurgical conditions (annealed, outgassed and formed to designed dimensions). Charging has been maintained continuously for over a month (some longer). No unexplainable heat output has occurred in our cells. One cell with an electrode that had been pre-deuterated has shown a small but significant increase in Tritium (4 σ affect). This might be explained by tritium contamination in the D₂ gas used for charging. This is under investigation. Samples are presently being tested which are similar to that used by Texas A&M and Stanford. Presently no difference, when correctly measured, is observed between the heavy and light water cells. However, we do find a significant temperature difference in the cells, with constant current.

Data from "Fus.-Data"



I. MATERIALS USED FOR ELECTROCHEMICAL EXPERIMENTS (please complete one sheet for each experiment)

	CATHODES	ANODES (Corresponding)
MATERIAL	Pd	Pt mesh baskets
PURITY	5'-9's	and Pt wire and foil
ALLOYING ELEMENTS	-----	

SOURCE OF MATERIAL	Johnson-Matthey
-----------------------	-----------------

PREPARATION CAST OR WROUGHT ANNEALED ATMOSPHERE VACUUM SPECIAL TREATMENT	vacuum & high purity Ar etched before insertion
---	--

CHARACTERIZATION STRUCTURAL CHEMICAL BEFORE OR AFTER USE METHODS RESULTS	TEM Scanning Auger Microprobe very brittle
---	--

NOTABLE
OBSERVATIONS

D / METAL RATIO ATTAINED

EXPERIMENT YIELDED HEAT	-----yes	x-----no
NEUTRONS	-----yes	-----no
TRITIUM	-----yes	-----xno
HELIUM	-----yes	-----no

II. MATERIALS USED FOR GASEOUS CHARGING EXPERIMENTS (please complete one sheet for each experiment)

MATERIAL

PURITY

ALLOYING
ELEMENTS

SOURCE OF
MATERIAL

PREPARATION
CAST OR WROUGHT
ANNEALED
ATMOSPHERE
VACUUM
SPECIAL TREATMENT

CHARACTERIZATION
STRUCTURAL
CHEMICAL
BEFORE OR AFTER USE
METHODS
RESULTS

NOTABLE
OBSERVATIONS

D / METAL RATIO ATTAINED

EXPERIMENT YIELDED HEAT
NEUTRONS
TRITIUM
HELIUM

-----yes
-----yes
-----yes
-----yes

-----X-----no
-----X-----no
-----X-----no
-----no

MATERIALS USED IN COLD FUSION EXPERIMENTS

PRINCIPAL INVESTIGATOR: Harold Wiesmann

ORGANIZATION: Brookhaven National Laboratory/Department of Applied Science

SHORT DESCRIPTION OF TYPE OF EXPERIMENT:

A search for steady state "excess" heat, neutron and tritium production from palladium cathodes electrolytically charged with deuterium was conducted.

RESULTS AND COMMENTS:

Four different electrolytic solutions were mixed and a different solution was used in each cell. The anodes were fabricated from platinum wire and two different palladium cathode sizes were employed. The four cells were initially charged at 60 ma/cm² for 192 hrs. Cell temperatures and the neutron count rates were monitored for an increase above the background level but no increases were observed. Geiger counters were used to periodically check for any indications of gamma emissions but the results were negative. It should be emphasized that a continuous record of the cell temperatures was not kept. Cells temperatures were recorded every 24 hrs. but unobserved temperature excursions may have occurred. On the assumption that longer charging times are required to initiate fusion, a current of 35-ma/cm² was applied for 624 hrs. Cell temperatures and neutron emissions were again monitored during this time. At the end of this extended charging period, the current was increased in three steps, 70 ma/cm², 105 ma/cm² and 140 ma/cm². Each current step lasted for 24 hrs. to allow the temperatures in the cells time to stabilize. This process was repeated but an additional current step to 175 ma/cm² was added. The attached graph is the data, (open square) for the first experiment where the current was stepped in 0.3 ampere increments. The abscissa is the power applied to the cell, E^*I , minus the enthalpy of gaseous deuterium and oxygen ($U=1.54$ V) and the ordinate is the change in temperature of the cells. The filled squares are data for a thermal calibration cell. An upper bound on the excess heat is 0.32 watts/cm³ for the largest Pd cathode. The neutron detection system has a calibrated efficiency of 0.028% (± 0.005) and no increase in neutron emission was detected as the current in the cells was increased. Tritium concentration increase up to 50% was observed. The cells, however, were run in the open configuration and the increase in tritium can probably be explained by preferential electrolytic concentration.

RECEIVED

AUG 15 1989

DEPT. OF PHYSICS

I. MATERIALS USED FOR ELECTROCHEMICAL EXPERIMENTS (please complete one sheet for each experiment)

	CATHODES	ANODES (Corresponding)
MATERIAL	Pd	Pt
PURITY	99.95	>99
ALLOYING ELEMENTS	none	none
SOURCE OF MATERIAL	Aesear	unknown
PREPARATION		
CAST OR WROUGHT	X	
ANNEALED		
ATMOSPHERE		
VACUUM		
SPECIAL TREATMENT		drawn
CHARACTERIZATION		
STRUCTURAL	no characterization performed	
CHEMICAL		
BEFORE OR AFTER USE		
METHODS		
RESULTS		
NOTABLE OBSERVATIONS	none	

D / METAL RATIO ATTAINED

EXPERIMENT YIELDED HEAT	-----yes	-X-----no
NEUTRONS	-----yes	-X-----no
TRITIUM	-----yes	-X-----no
HELIUM	-N/A---yes	-N/A---no

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IN REPLY REFER TO:
6110-478:CTW:bmd
11 September 1989

Dr. J.R. Huizenga, Co-Chairperson
ERAB Cold Fusion Panel
U.S. Department of Energy
1000 Independence Avenue, S.W.
Washington, D.C. 20585

Dear Dr. Huizenga:

In response to your request to Dr. Mintmire, please find enclosed a brief summary of research on "cold fusion" carried out by the Theoretical Chemistry Section at NRL. Also please find enclosed a reprint of some of our work on this topic that has appeared in Physics Letters A.

If we can be of any additional assistance to you, please do not hesitate to contact us.

Sincerely,

A handwritten signature in black ink, reading "Carter T. White", is positioned above the typed name.

CARTER T. WHITE, Head
Theoretical Chemistry Section
Chemical Dynamics & Diagnostics Branch
Chemistry Division/Code 6119

Enclosures

Summary of Research on Cold Fusion

J.W. Mintmire, B.I. Dunlap, D.W. Brenner
R.C. Mowrey, and C.T. White

Theoretical Chemistry Section
Code 6119
Naval Research Laboratory
Washington, DC 20375-5000

Our research has been primarily devoted to investigating one suggested mechanism for cold fusion from a theoretical approach. We had read the March 24 Wall Street Journal article at the March APS meeting in St. Louis announcing the work of Pons and Fleischmann, and the next week back at NRL we gathered what preprints and newspaper articles we could on this subject. Reviewing this material we recognized that piezonuclear fusion, as discussed by Jones et al., required that the repulsion between deuterons in gas phase D_2 be somehow severely reduced in the metal lattice. The members of our research group possessed a broad range of skills that were particularly appropriate to theoretically examine this requirement.

Using established first-principles and empirical methods, we studied the effective interaction between deuterons in a palladium lattice. The embedded-atom method was used to examine the effective interaction between two deuterons in the neighborhood of both the octahedral and tetrahedral interstitial sites of the Pd lattice. First-principles local-density functional calculations were carried out on model palladium clusters for both the octahedral site and the tetrahedral site, with Pd_6H_2 the largest cluster studied. Ab initio Hartree-Fock calculations were also carried out on Pd_3H_2 and PdH_2 clusters.

Piezonuclear fusion of deuterons, as discussed by Jones, requires a sharp decrease in the equilibrium inter-deuteron separation, and/or a sharp reduction in the repulsive potential between deuterons in the metal lattice. All our calculations implied that neither possibility would occur in the palladium-deuterides. Local-density functional calculations on titanium-deuteride clusters led us to similar conclusions for that system. Our palladium-deuteride results were published as a preliminary note in Physics Letters A 138, 51-54 (1989) [copy enclosed] with more detailed descriptions of our work in press in the Journal of Fusion Energy, and submitted for publication to the Journal of Chemical Physics. Similar results (describing local-density functional, Hartree-Fock, or embedded-atom calculations) were subsequently published by a number of other workers with essentially the same conclusions as ours.

understanding that all such results are directly applicable to deuterium.

In our condensed-phase studies, energies and geometries are calculated using the embedded-atom method [4], an empirical potential approach. The potential functions for Pd and H are those reported by Daw and Baskes [4], with the potential functions for Pd determined by fitting to the lattice spacing, elastic constants, sublimation energy, vacancy formation energy and the binding energy difference between the bcc and fcc phases. The functions for hydrogen were determined by fitting to the embedding energies calculated by Puska et al., [5], and the heat of solution for H in Pd. Using these potentials, Daw and Baskes [4] have calculated a migration energy for H in Pd of 0.26 eV, and a lattice expansion of 4% upon formation of the PdH hydride. Both numbers are in agreement with the experimentally determined values of 0.26 eV and 3.5% (for PdH_{0.6}), respectively. These potentials have also been shown to predict subsurface occupation and phase changes for hydrogen adsorbed on Pd(111) [6].

The bulk crystal is modeled by 108 Pd atoms arranged in an fcc structure with periodic boundary conditions applied in all directions. Minimum energy structures were determined by relaxing the positions of all atoms using a direct minimization technique [7]. The cell boundaries were maintained at the 4% expansion of the hydride over pure Pd. In an attempt to locate a site within the Pd lattice where two hydrogen atoms might be held closely together, calculations were performed which differed in the number and initial positions of hydrogen atoms within the lattice. For hydrogen/palladium ratios less than one, the hydrogen atom prefers the octahedral interstitial site [8]. Therefore, we first considered two atoms in this site.

All of the octahedral interstitial sites within the crystal were first filled with H atoms. An additional H atom was then randomly placed in the vicinity of a filled octahedral site, and the system was allowed to relax. Ten positions were tried, and in each case the hydrogen atoms repelled each other and migrated towards opposite faces of the surrounding octahedral cell. The relaxed H-H bond distances varied from 1.68 to 1.35 Å, with no minima found at shorter distances. Next, the same PdH structure was used, but with one octahedral site occupied with three

hydrogens this time instead of two. Again the hydrogen atoms repelled one another and no potential energy minima were found for H-H bond distances shorter than 1.4 Å. The shortest H-H bond distances that we found occurred for the local configuration drawn in fig. 1a in the octahedral cage. The two relevant hydrogen atoms are darkened in the figure and the others (unshaded) occupy the remaining octahedral sites. As can be seen in the figure, the two hydrogen atoms repel each other along the diagonal of the cubic unit cell.

These results using empirical potential methods led us to carry out linear-combination-of-Gaussian-type-orbitals local-density functional (LCGTO-LDF) calculations [9] ($X\alpha$ with $\alpha=0.7$) on a D_{3d} -symmetric Pd₆H₂ cluster. This cluster contains the six equivalent palladium atoms that are at the faces of a cube surrounding the octahedral interstitial region. The hydrogen atoms were constrained to lie symmetrically along the diagonal of the cube. The Pd positions were fixed at the pure palladium fcc lattice geometry. The Pd orbital basis set was a 17s/11p/8d basis [10] and the H orbital basis set was a 6s basis [11]. Both basis sets were augmented with a polarization function of exponent 1.0 bohr⁻². All exponents greater than 10.0 bohr⁻² were contracted according to an atomic calculation. Basis sets for fitting the potential were derived from these orbital bases [9]. Several variations on this approach to selecting basis sets for fitting the potential all give nearly identical isomerization energies for 13-atom first-transition-series clusters [12]. The calculations are nonrelativistic. Relativity is marginally important in describing the electronic structure of totally symmetric octahedral and tetrahedral clusters containing a hydrogen atom [13].

Within this eight-atom cluster model the H-H in-

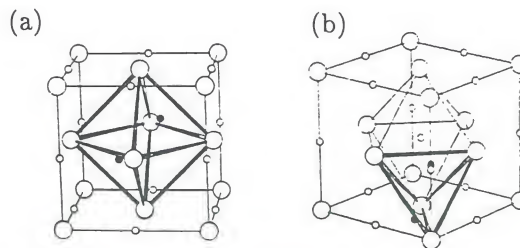


Fig. 1. Hydrogen pair in hydrogenated fcc palladium lattice at (a) octahedral and (b) tetrahedral interstitial sites.

CHEMICAL FORCES ASSOCIATED WITH DEUTERIUM CONFINEMENT IN PALLADIUM

J.W. MINTMIRE, B.I. DUNLAP, D.W. BRENNER, R.C. MOWREY, H.D. LADOUCEUR,
P.P. SCHMIDT, C.T. WHITE and W.E. O'GRADY*Naval Research Laboratory, Washington, DC, USA*

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First-principles and empirical methods are used to study the effective interaction between two deuterons in a palladium lattice. No effects are found to suggest confinement of deuterons at distances much smaller than the gas-phase D_2 separation.

Fleischmann and Pons [1] have recently presented evidence for the nuclear fusion of deuterium induced by electrochemical means in palladium at ambient temperatures; additional work by Jones et al. [2] provides further support for this effect. At room temperature, where the kinetic energy of the nuclei is not a dominant factor, the fusion rate is strongly dependent on the equilibrium distance between the two nuclei [3]. The nuclear tunneling probability through the barrier introduced by internuclear Coulomb repulsion is such a sensitive function of this distance that fusion in liquid deuterium is negligible whereas muon catalyzed fusion occurs in 10^{-9} seconds because the nuclei are approximately 200 times closer together. Interpolating between these two rates, Jones et al. [2] suggest that the apparent fusion rate can be achieved by the "squeezing" of deuterons in the metal lattice to a separation on the order of ~ 0.3 Å. These authors speculate that such a confinement [2] might be achieved by quasi-electrons with large effective mass in the deuterated lattice. Screening of the bare deuterium-deuterium nuclei electrostatic repulsion requires variation of the electronic charge density on an even smaller scale. The uncertainty principle alone associates a high kinetic energy with this degree of localization. If "heavy" quasiparticles do not provide the mechanism for such deuteron confinement, then the question is raised of whether compressive forces exerted by the metal structure could lead to such a confinement of deuteron pairs at the neces-

sary close proximity. Such confinement could conceivably be produced either by producing a minimum energy in the potential energy surface for a short D-D distance, or by screening effects from the metal charge distribution that substantially weaken the repulsive D-D internuclear potential.

To explore the possibility of such a mechanism, we have performed a set of model studies of the energetics of the deuterium-deuterium potential in a palladium crystal. One major objective has been to estimate, within the Born-Oppenheimer approximation, the effective interaction between two deuterons in a palladium lattice, at scales of 0.1–1.0 Å. We used embedded-atom empirical potential methods to study the effects of the periodic lattice on deuterium interstitials and used both local-density functional (LDF) and Hartree-Fock (HF) first-principles cluster calculations to study the energetics of deuterium nuclei in finite palladium clusters. Using these empirical and quantum chemical calculations we have sought, but not found, a position in the lattice where the deuterium pair has the unusually short internuclear separation that has been speculated as necessary for fusion [2]. These studies partially quantify the energetics of the deuterium-deuterium interaction in crystalline palladium. Because all of these results for electronic effects are based on the Born-Oppenheimer approximation and thus independent of nuclear mass, we will henceforth discuss all potentials with respect to hydrogen, with the

teraction energy in an octahedral site is tabulated in table 1. For our cluster calculations we define the H-H binding energy to be the total energy of a pair of hydrogen atoms in the cluster minus twice the energy of a single hydrogen at the symmetric position plus the total energy of the empty cluster. As table 1 directly indicates, two hydrogen atoms repel each other quite strongly in the octahedral site. Furthermore, for short internuclear separations the H-H repulsive interaction is significantly stronger than in the gas phase.

We also considered the case where two hydrogen atoms are in a single tetrahedral interstice. As before, the embedded-atom method calculations started with hydrogen atoms centered in all octahedral sites in the Pd lattice, producing a lattice with stoichiometry PdH. Two additional H atoms were then placed in one tetrahedral cell and ten sets of calculations were run using different starting positions for the pair of hydrogens. The relaxed H-H distances ranged from 1.32 to 1.60 Å. Then five trials were run where three atoms were introduced into one tetrahedral site. In every case the H atoms again repelled each other to opposite sides of the interstice, just as occurred for the octahedral interstice, but now, the closer of the three H-H distances varied from 1.27 to 1.63 Å. A representative case of the short bond distances in the tetrahedral cage is drawn in fig. 1b. In this figure, the two relevant hydrogen atoms are shaded. The other hydrogen atoms in the figure lie at the centers of the octahedral interstices as before.

Table 1
Relative LDF H-H potentials: gas phase and in the octahedral site (Pd_6H_2).

$R_{\text{H-H}}$ (Å)	Octahedral (hartrees)	Gas phase (hartrees)
2.117	0.02464	
1.852	0.02553	
1.588	0.02870	
1.323	0.03510	
1.058	0.04924	0.02771
0.953	0.05959	0.01308
0.847	0.07338	0.00251
0.741	0.09378	0.00029
0.635	0.13322	0.01416
0.529	0.20182	0.05957
0.423	1.33286	0.16978

Both hydrogen atoms end up being very close to the plane of three palladium nuclei, as can be noticed most readily for the lower-left hydrogen in fig. 1b. From the other darkened hydrogen atom in the figure it is apparent that both hydrogen atoms lie off the center of the triangular face towards the center of a nearest-neighbor palladium bond. Therefore, we chose to do LCGTO-LDF calculations for two geometries. If the hydrogen atoms went through the center of two faces the relevant Pd_4H_2 cluster has C_{2v} symmetry. If the hydrogen atoms went to the center of opposite bonds the cluster would have D_{2d} symmetry. The results are given in table 2 and again we find strong repulsive forces. However, we do find a minimum in the forces and a bound H_2 complex in the D_{2d} symmetry, in large part because of the additional steric hindrance introduced by forcing the hydrogen nucleus directly through the Pd-Pd bond.

The above empirical and quantum chemical calculations show that two hydrogen atoms in either the octahedral site or the tetrahedral site repel each other towards the boundaries of the interstitial cells. We therefore considered two hydrogen atoms approaching each other symmetrically about the three-palladium-atom boundary between these two sites. Calculations of the total energy of a Pd_3H_2 cluster were performed using the LCGTO-LDF method using the above basis and with an ab initio Hartree-Fock approach using an STO-3G basis set [14]. The complex was placed in a trigonal bipyramidal arrangement with the hydrogen atoms occupying the apexes of the pyramids. The distance of the hydrogen atoms from the plane of the palladium atoms was varied

Table 2
Relative LDF H-H potentials in the tetrahedral interstitial site (Pd_4H_2).

$R_{\text{H-H}}$ (Å)	D_{2d} (hartrees)	C_{2v} (hartrees)
1.588	0.08051	
1.270	0.07414	
1.058	0.07372	
0.953	0.07723	
0.847	0.08480	0.06338
0.741	0.10033	0.08168
0.635	0.13212	0.11587
0.529	0.19432	0.18084
0.423		0.30867

and the Pd-Pd bond length was held fixed at 2.75 Å. The results of these calculations of the relative LDF energy (defined above) and of the total HF energy are given in table 3. They demonstrate that the total energy of the system increases as the separation between the hydrogen atoms decreases in both local-density functional and HF. This position is close to the maximum barrier for diffusion of H in Pd, 0.26 eV. Therefore 0.5 eV should perhaps be added to the repulsive energy for close approaches.

On a still smaller scale, we performed HF PdH₂ calculations to see whether confinement or collisions within the Pd 4d electronic shell might yield a relatively low-energy conformation of two deuterium nuclei near a palladium nucleus. Again all repulsions were greater than without the palladium atom.

Our calculations suggest that at short distances the H-H forces in the palladium crystal are more strongly repulsive than in free molecular hydrogen, and that the mutual repulsion of the two hydrogen atoms within an interstitial will cause them to maximize the distance separating them. Both effects should decrease rather than increase the probability for fusion in the lattice over that in free D₂. The concentration of H atoms in PdH is nearly the same as that for solid hydrogen at 4 K. The separation between octahedral sites in the PdH lattice, however, is 2.86 Å, while because of its molecular nature solid hydrogen has a shorter separation of 0.74 Å between hydrogens in each pair. This again suggests that based on chemical forces the probability for fusion should be less in the metal than in solid hydrogen. Finally, we note that

Table 3
Pd₃H₂ cluster H-H potentials in LDF and HF.

R_{H-H} (Å)	Relative LDF energy (hartrees)	Total HF energy (hartrees)
1.693	-0.02280	
1.482	-0.02110	
1.270	-0.01248	
1.058	0.00308	
0.953	0.01415	
0.847	0.02907	
0.741	0.05248	-14668.68762
0.688		-14668.66878
0.635	0.08996	-14668.65495
0.529	0.15606	
0.423	0.28568	-14668.57368

experimental evidence [15] suggests that the stoichiometry of the palladium hydrides for which appreciable fusion is detected may be PdH₂. Calculations using the embedded-atom method described above, in which the dimensions of the cell were allowed to change, showed an additional average expansion of the lattice parameter of 9.5% for PdH₂ over PdH. Hence, the separation between an octahedral site and an adjacent tetrahedral site is 1.9 Å, a distance which is still too large for appreciable fusion to occur. These calculations suggest that confinement of deuterium within the Pd lattice resulting from chemical forces is not responsible for the enhanced fusion of deuterium observed in palladium.

We wish to thank the Research Advisory Committee of the Naval Research Laboratory for a grant of computer time which was used in performing a portion of these calculations.

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AT&T Bell Laboratories

600 Mountain Avenue
Murray Hill, NJ 07974-2070
201 582-3000

September 19, 1989

John Huizenga
Professor of Nuclear Chemistry
Department of Chemistry
University of Rochester
Rochester, NY 14627

Dear Professor Huizenga:

In response to your request on materials related to the cold fusion calorimetry, I am sending you our paper in this area. The enclosed manuscript "Calorimetric Studies of Electrochemical Incorporation of Hydrogen Isotopes into Palladium" will be presented in The Electrochemical Society Fall Meeting and will be submitted for journal publication.

Yours truly,

A handwritten signature in cursive script, appearing to read "Henry H. Law".

H. H. Law
Supervisor
Electrochemical Processes Group

Enclosure

Copies with enclosure to:

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CALORIMETRIC STUDIES OF ELECTROCHEMICAL INCORPORATION OF HYDROGEN ISOTOPES INTO PALLADIUM

J. W. Fleming

H. H. Law

J. Sapjeta

P. K. Gallagher

W. F. Marohn

AT&T Bell Laboratories
Murray Hill, New Jersey 07974

ABSTRACT

The formation of palladium hydride and deuteride by electrolysis has been studied with high precision calorimetry using sealed cells with in-situ recombination of gaseous products as well as open cells. Palladium electrodes prepared by different methods were studied. With sealed cells of our design in the heat flow isothermal calorimeter, the uncertainties associated with the gas evolution, evaporation, entrainment, and side reactions were effectively eliminated. No unexpected excess heat was observed within energy balance error of about 2%.

CALORIMETRIC STUDIES OF ELECTROCHEMICAL INCORPORATION OF HYDROGEN ISOTOPES INTO PALLADIUM

*J. W. Fleming
H. H. Law
J. Sapjeta
P. K. Gallagher
W. F. Marohn*

AT&T Bell Laboratories
Murray Hill, New Jersey 07974

INTRODUCTION

Observations of excess heat evolved from the electrochemical incorporation of hydrogen isotopes into palladium have been recently reported (1). The investigators have linked this heat with nuclear fusion of the hydrogen isotopes. However, their calorimetric experimental conditions were based on certain assumptions about the nature of the electrochemical process which could lead to significant errors in the interpretation of calorimetric results.

We have developed an electrochemical calorimetric apparatus to provide unambiguous experimental conditions for the observation of such excess heat. A novel sealed cell has been constructed in which no by-product of the electrochemical process is lost to the environment without being accurately detected. With this apparatus and extensive calibration of the calorimeter, we are able to account to within 2 % for the heat associated with the electrochemical incorporation of hydrogen isotopes into palladium.

When the electrolysis of water is performed using a platinum anode and a palladium cathode, oxygen gas is generated at the platinum surface, while hydrogen is evolved at the palladium. Most of the hydrogen initially formed is incorporated into the palladium cathode. As the hydrogen content increases, hydrogen gas evolves from the surface. The formation of gaseous products complicates the interpretation of the calorimetric data. Sherfey and Brenner (2) reported the difficulties of accurately measuring the heat of reaction for the electrolysis of water. Their main concern, supported by the work of Joncich and Hackerman (3), was the recombination of oxygen and hydrogen in the electrolyte during the electrolysis. In addition, other energy losses, such as the evaporation and entrainment of the electrolyte by the gas being evolved, could lead to erroneous calculations and conclusions. In the present work, we employed our precision calorimeter to address the subject of the excess heat observed in the incorporation of deuterium in palladium.

EXPERIMENTAL

Cell Design and Procedure

Both open and sealed cells were used in this work. The electrolytic cell was a Pt tube with an inner diameter of 1 cm and a length of 7.5 cm. The Pt container itself served as the anode for the experiments. The cathode was positioned along the axis of the cell with Teflon spacers that assured proper orientation. The cathode lead for all experiments was 99.95% Pt wire (0.78 mm dia.). Teflon spacer design prevents exposure of the Pt to the solution phase. Specimens for test were cut to the desired length and spot welded to this lead. The open cells, shown in Fig. 1, were placed in 50 cm fused silica tubes designed to carry away the vapors evolved from the electrochemical process. The sealed cells, shown in Fig. 2, were placed with copper sleeves in alumina tubes, which functioned to insure better thermal contact with the heat detection assembly. The sealed cell also had a mesh of fine Pt wire (0.25 mm dia.) above, and separated from, the lower region containing the electrolyte, to serve as a recombination catalyst for the products of the reaction, H_2 , HD or D_2 , and O_2 . The mesh was separated from the cathode by a fused silica tube surrounding the cathode. The cell was sealed with a Teflon stopper machined to fit the Pt walls very snugly. The cathode lead ran through the center of this plug and also fit tightly. The seal would hold up to an excess pressure of 3000 torr.

To ensure that the catalyst area was sufficient for recombination, a sealed cell was modified to allow pressure monitoring during electrolysis. A capacitance-type transducer (MKS Instruments, Type 122, 0.15% accuracy) was used for the pressure measurement. The mesh of fine Pt wire with a surface area of about 40 cm^2 was adequate to allow a sustained electrolytic current of 250 mA without excessive pressure buildup. When the samples were run in the calorimeter, the seal was verified by weight measurements. Measurements were made to $\pm 0.01\text{ g}$ sensitivity.

Experiments were performed with several types of Pd and Pt cathodes. The Pd electrodes varied in size and microstructure. The diameter of the Pd wire specimens ranged from 0.5 to 3 mm. The electrodes were either used as received from an existing wire spool, melt cast wires with/without cold work, or hot pressed Pd powder. All electrodes were cleaned by evolving oxygen for 15 seconds in an alkaline cleaner (Enbond Cold Action Cleaner, Enthone Inc.), rinsing in deionized water, dipping in 6 M HCl for 15 seconds, rinsing

in deionized water and drying. A vacuum heating of the metal was employed in many experiments to remove any hydrogen present prior to electrolysis.

The experiments were performed as follows. The calorimeter (see below) was equilibrated to room temperature (about 20°C) and held isothermally for the duration of each test. The maximum variation in the room temperature was 2°C. Apparatus temperature was monitored continuously for the entire experiment. With the typical power input being less than 2 W, no increase in the temperature of the calorimetric alumina block was detected. The electrolytic cells were placed in the calorimetric cells with the anode and cathode leads exiting from the top opening of the chamber. Samples were run either with reference cells with all Pt electrodes or with standardized Joule heaters placed in the second calorimetric chamber. In many differential experiments the same sample was run alternately in both calorimetric chambers with same result, which demonstrated that there is no difference in sensitivity between the two chambers. Most sealed cell experiments were run with a single cell in one chamber.

Experiments were run for times varying from less than 1 to greater than 40 days. All the experiments were run in the constant current mode using Princeton Applied Research Galvanostats (Model 362 or 363). The accuracy of the current control was better than 0.1%. The electrolyte was either 0.1 M LiOH in H₂O or 0.1 M LiOD in D₂O. The LiOD electrolyte was prepared by dissolving an appropriate amount of Li₂O (Alfa Products, 95%) in D₂O (Aldrich Chemicals, 99.9%). In the early phase of this investigation, the calorimetric output was measured using the Astra Scientific International thermal analysis software running on an AT&T personal computer Model 6300 with data stored at intervals of 5 to 90 seconds. The cell voltage was monitored with a Nicolet Digital Oscilloscope Model 2090 with a precision of 2.5 mV and an accuracy of better than 0.2%. The cell voltage was measured at intervals of 5 to 20 seconds. The power input was the product of the cell voltage and the current. To ease the burden of data reduction, a Hewlett Packard data logger Model 3497A was eventually used to measure both the calorimetric output and the cell voltage simultaneously. The data were stored and analyzed using a Basic program running on an AT&T personal computer Model 6300.

Thermogravimetric analysis was performed on selected specimens to determine the amount of deuterium incorporated into the palladium after the calorimetry experiment had been completed.

Thermogravimetry was performed using the Perkin-Elmer System 7 with the standard furnace. A heating rate of 20°C/min was used in an atmosphere of flowing nitrogen. Buoyancy and aerodynamic forces were compensated by subtraction of a run made without a sample under the same experimental conditions. In calculating the composition, the weight loss was attributed entirely to deuterium.

Calorimeter

The Setaram HT 1000 calorimeter used in this study functions as a heat flow isothermal calorimeter. As shown in Fig. 3, the sample is in direct contact with the calorimetric chamber and is equilibrated to its temperature. There are two cell chambers, each 1.7 cm in diam. and 8 cm in length. The useful calorimetric cell volume is 15 cc. The measured calorimetric output is the thermal power which flows into or out of the electrolytic cell to the thermostated chamber. The heat flow detector consists of a multitude of identical thermocouples connected in series and oriented around the sample (Fig. 4, 22 ring arrays of 36 thermocouples) such that it is reasonable to assume all the heat flowing from the sample must pass through the thermocouples. Calibration is discussed below. Since the sample is at thermal equilibrium with the surrounding chamber, the enthalpies of reaction are measured directly, and it is not necessary to know the heat capacity of the sample under observation. Isothermal measurements can continue for very long times and the calorimeter is thus applicable to both slow and fast reactions.

Two completely symmetrical calorimetric heat flow detectors are connected in opposition and seated symmetrically in the calorimetric unit. This provides autocompensation for thermal instability and results in better resolution of the differences in the two cell compartments. Total outputs of each compartment are also available. The calorimetric detectors sit within and attached to an alumina block which serves as a heat source or sink depending on the thermal response of the sample. For the differential experiments, the resolution of power detection was about 10 μ W. In later experiments with a single electrolytic cell, sensitivity was reduced to 2.5 mW when the power input was about 2 W.

Calibration

The system was calibrated using Joule heating from a precision resistor placed in the calorimetric chamber. A constant current was put through the resistor for a given time and the output integrated to yield the energy released. From this the power response function of the calorimetric chambers was determined.

Several variations of this procedure were used to accurately understand the response of the apparatus. In one series of calibrations the precisely calibrated Joule heater was placed in the calorimetric chamber in conjunction with a second Joule heater. In this configuration the response of the unit was checked as a function of baseline energy output. A 100 mW signal from the precision Joule heater yields identical responses of 100 mW at baseline levels of 20, 500 and 1000 mW, as shown in Fig. 5. This is an important distinction from various forms of non-isothermal calorimetry in which the cooling constant of the system varies with total power input.

In another series of experiments a specific Joule heater was equilibrated at a given power level and switched between cells to compare the response of the reference and sample cells. Here a baseline variation of approximately 4 mW was observed. However, the total energy released was measured to be the same to within 0.4 % regardless of the level tested. The maximum calibrated power range for each cell was 4 W.

In another set of calibrations a standardized Joule heater of about 0.9 cm length and 0.2 cm diameter was placed at different depths within the sample cell having essentially the same electrode configuration and leads as employed in the actual experiments. This assembly was placed in the calorimetric chamber and the heat was observed as a function of the resistor's vertical position. The response to the same load at different depths and different loads at the same depth was measured using the power pulse integration method. In these experiments we observed that the lower end of the cell gave a calorimetric signal approximately 2 % higher than the average value of the cells and the top measurements were approximately 2 % lower. This variation may be systematic or due to differences in the heat conducted away from the sample chamber through the power leads. This variation needs to be considered in the electrochemical calorimetric experiments because there is a geometric non-uniformity in the cell responses. Joule heating occurs towards the bottom and recombination of hydrogen and oxygen occurs at the top of the cell.

RESULTS & DISCUSSION

In early experiments differential runs were made with an electrolytic cell in each calorimetric compartment which had the same cell construction with the exception of cathode metal or electrolyte. Such experiments permitted running the calorimeter at highest sensitivity (10 μ W). In addition, assumptions about the electrochemical processes can be relaxed in interpreting the data. In calculating the excess heat,

Pons et al. (1) assumed no recombination of oxygen and hydrogen or deuterium. In contrast, if one assumes the same degree of reaction occurring in both cells, e.g., the same degree of recombination and the same evaporation losses, differential runs of palladium and platinum electrodes could yield meaningful results.

Table I summarizes the differential runs. The 1.25 mm diameter palladium electrode was from an available wire spool (99.95%). With the open cells, the electrolyses were run for 15 to 25 hours. There was no excess heat generated and the balance between the heat detected and the power input (cell voltage x current) was within 50 mW, which is about 5 to 10 % of total power input. In these open cell experiments, it was also visibly evident that there was condensation on the external wall of the Pt container (anode). The liquid was either from the recombination of the gases or from gas entrainment of electrolyte. Since the pH of the condensate was acidic (about 4), the gas entrainment is unlikely. Also, the relatively low experimental temperature would make significant evaporation of solvent improbable. Therefore, the liquid is predominantly the product of the gaseous recombination.

The observed condensation phenomenon and the uncertainty level in the power balance encouraged us to develop a sealed cell by incorporating a mesh of fine Pt wires for the recombination of gaseous products. With sealed cells, the uncertainty associated with the recombination, whether it is in the liquid phase as reported (2-3) or in the gas phase as we observed, would be eliminated. For the long time runs, it would also remove the influence of impurities accumulated from the continuous addition of D₂O. There is no heat loss due to evaporation or entrainment. It appears the sealed cell design is most suitable for detecting heat imbalance.

The possible complication from the sealed cell design would be the extent of side reactions controlled by pressure effects in the gas phase. To estimate the extent of this uncertainty, the pressure was monitored during electrolysis. Figure 6 shows the initial pressure buildup with time. The pressure increase of 50 torr in the first 15 minutes is consistent with the accumulation of unreacted oxygen while the deuterium is incorporated into palladium. The pressure increased as the current density was increased, but the maximum pressure was less than 300 torr at 1 A/cm². In addition, the degree of the deuterium incorporation was comparable to that for the open cells for the same time duration. The amount incorporated in longer

electrolysis experiments was typically PdD_x ($0.45 < x < 0.75$).

With sealed cells, differential runs are not as desirable because it is not possible to rule out the cell leakage problem. The cause of an imbalance of excess heat between two cells could not be determined precisely. Table 2 shows the conditions employed with a single sealed cell.

The experiments using Pt electrodes were performed to establish the standard response of the electrolytic cell and the calorimetric equipment. No excess heat (rate of heat flow from the electrolytic cell minus the power input) was measured for current densities ranging from 60 to 300 mA/cm^2 . Figure 7 shows the lack of an excess rate of heat generation at 100 mA/cm^2 . The steady state fluctuation indicated the dynamics of the electrolysis of the D_2O and the recombination of D_2 and O_2 . The constantly changing electrolyte level (bubble effects) would lead to the fluctuations of cell voltage and hence the power input. Figures 8 & 9 depict the initial and final responses of the calorimeter and the power input for the same experiment. The change of power input was instantaneous with the current, and yet the change of heat flow was much slower. Since the response time of the calorimeter is about 150 seconds, the initial difference between the power input and the heat flow had to be attributed to heating of the electrolytic cell and the formation of gaseous products. As expected, energy that had been stored in the system during electrolysis continued to be released for some time after stopping the current (Figure 9). The steady state excess rate of heat generation was about zero. The data were integrated to yield the complete energy balance: a total of 7633 J of energy was detected and a total of 7467 J of energy was input. The energy was balanced to within 2.2% positive, which is equivalent to an average of less than 3 mW excess rate of heat generation. Similar behavior was observed for electrolyses at 60 and 300 mA/cm^2 . The 2 % difference is mainly attributed to the geometric non-uniformity in the cell response of our system.

Identical responses were observed for palladium electrodes in sealed cells. Figure 10 shows the excess rate of heat generation of a 2 mm melt cast palladium electrode running at a power input level of about 1100 mJ/s after 14 days. At about 1300 minutes (on the 14th day), a 100 mW calibrating signal was sent to the precision Joule heater located in the second calorimetric chamber. This resulted in a 100 mW decrease of the calorimetric output, which confirmed the accuracy of the calorimeter response. The signal noise was mainly due to the change of the power input when the electrolyte level fluctuated. Fig. 11 shows the excess

rate of heat generation of a similar Pd electrode after 33 days of electrolysis and the total power input was about 1300 mJ/s. The excess rate of heat generation for these runs was also less than 2%.

The sealed cell runs showed agreement between input power and output power to within 2%. It is worth reiterating that no assumptions were required to achieve this result. The 2 % variation observed was explained by the response of the calorimetric cell detector to exothermic events in specific orientations. The instrument is calibrated for a heat integrated over the entire detector area. As reported above it was found that the lower portions of the cell, which are better insulated, detect about 2 % more heat than the average and that the upper sections detect about 2 % less than the average for the sample cell design employed in these experiments. This effect is sufficient to explain the difference between input and output power given the geometrical orientation.

SUMMARY

In the calorimetric study of the incorporation of hydrogen isotopes into palladium, the power input and output of the corresponding electrolytic cells were always matched to within 2%, whether the cathode was Pt or Pd (in any form). This conclusion followed from the development and application of a sealed cell with an in-situ Pt wire recombination catalyst. The uncertainties associated with the gas evolution, evaporation, entrainment, and side reactions are effectively eliminated.

Since the initiation of our work, Hayden et al. (4) reported their work on a calorimetric search for evidence of cold fusion using an in-situ palladium filament to recombine evolved gases. Their observation of no excess heat generation is in agreement with our results.

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FIGURE CAPTIONS

- Figure 1: Schematic of an open electrolysis cell.
- Figure 2: Schematic of a sealed electrolysis cell.
- Figure 3: Schematic of a dual-chamber Calvet Calorimeter.
- Figure 4: Heat-flow detector thermocouple arrangement.
- Figure 5: Calorimeter calibration with Joule heater.
- Figure 6: The initial pressure buildup of a sealed cell with a recombination catalyst; Pd electrode, 1.25 mm dia., 60 mA/cm².
- Figure 7: Excess rate of heat generation vs. time; .75 mm dia. Pt electrode; 100 mA/cm².
- Figure 8: Initial rate of heat generation and power input vs. time; .75 mm dia. Pt electrode; 100 mA/cm²; same experiment as Fig. 7.
- Figure 9: Final rate of heat generation and power input vs. time; .75 mm dia. Pt electrode; 100 mA/cm²; same experiment as Fig. 7.
- Figure 10: Excess rate of heat generation vs. time; 2 mm dia. cast Pd electrode; 1100 mJ/s.
- Figure 11: Excess rate of heat generation vs. time; 2 mm dia. cast Pd electrode; 1300 mJ/s.

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OPEN ELECTROLYSIS CELL

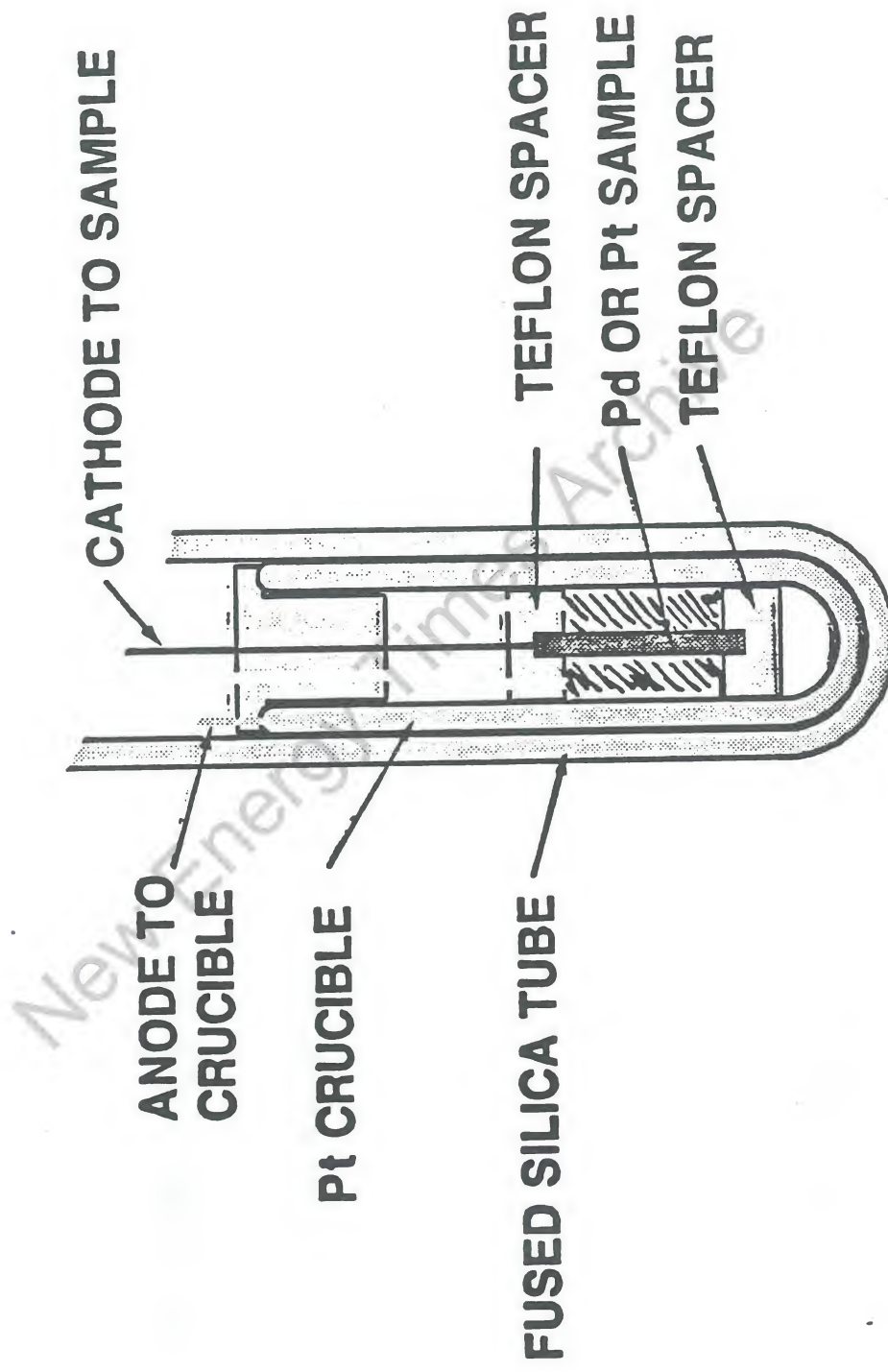


Figure 1

CLOSED ELECTROLYSIS CELL

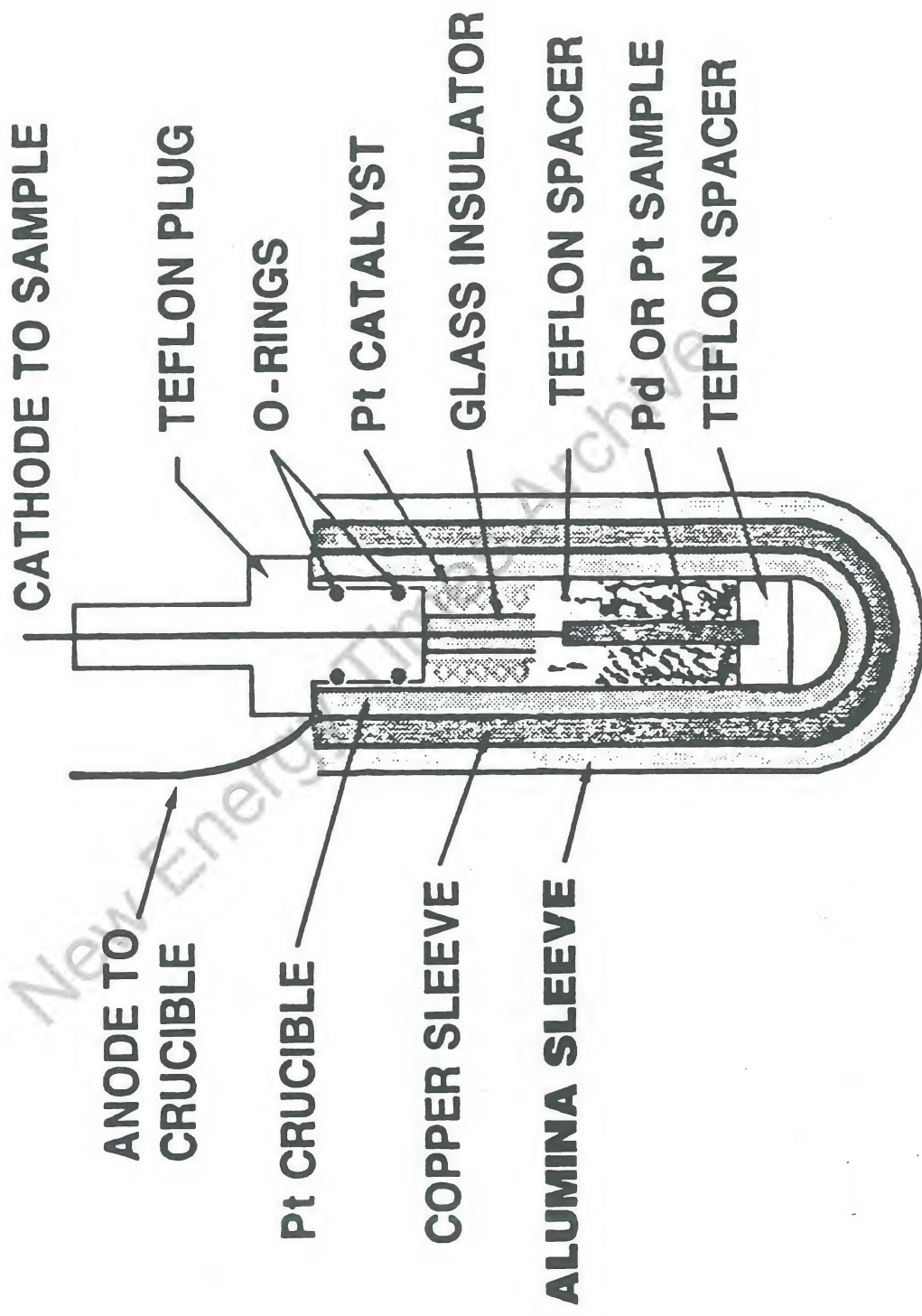


Figure 2

DUAL-CHAMBER CALORIMETER

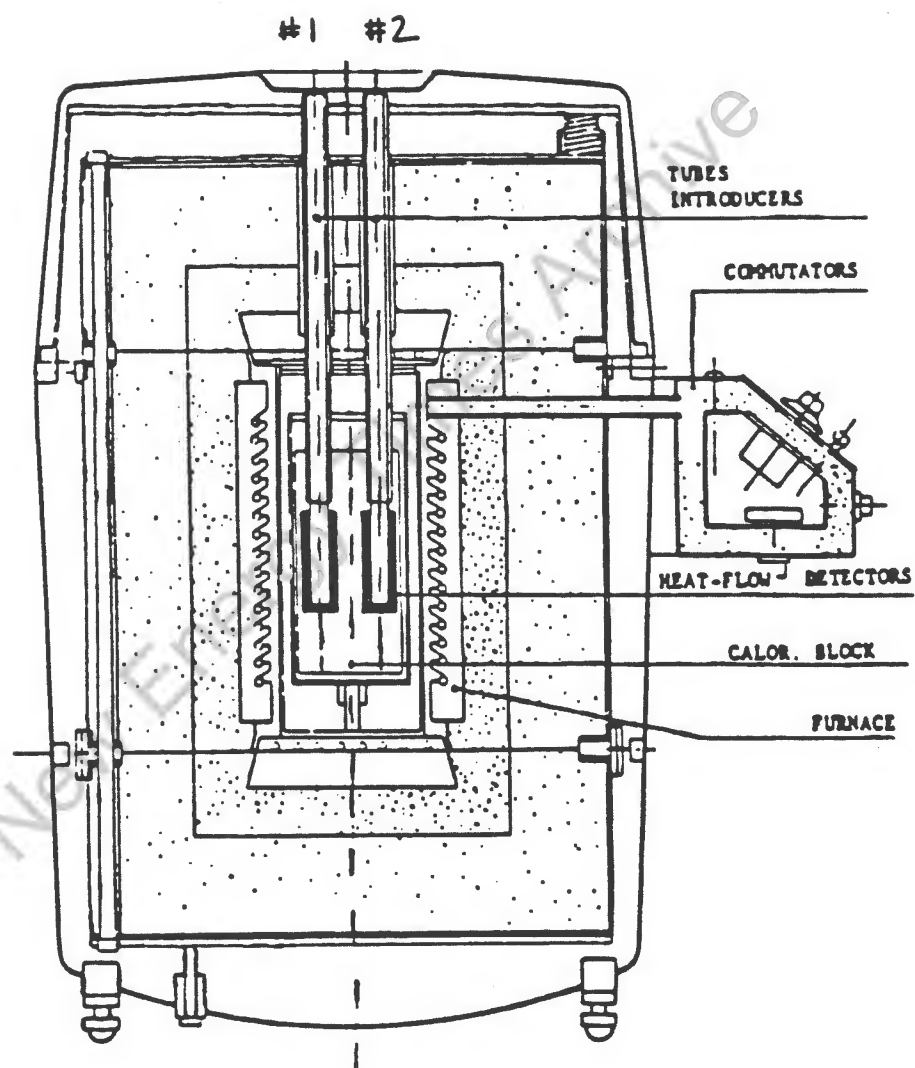


Figure 3

HEAT-FLOW DETECTORS

THERMOCOUPLES ARRANGEMENT

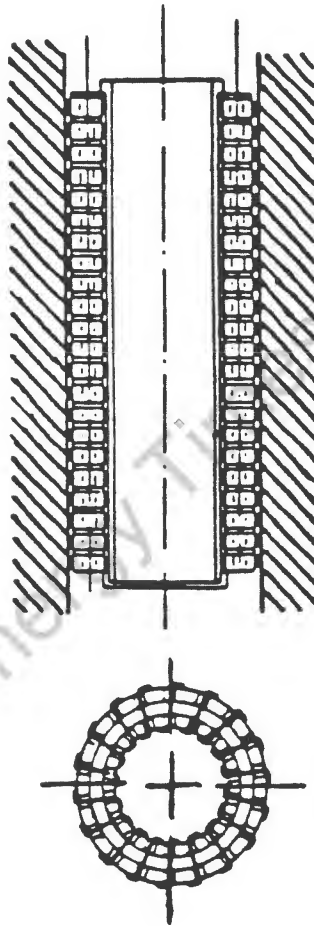


Figure 4

CALORIMETER CALIBRATION WITH JOULE HEATER

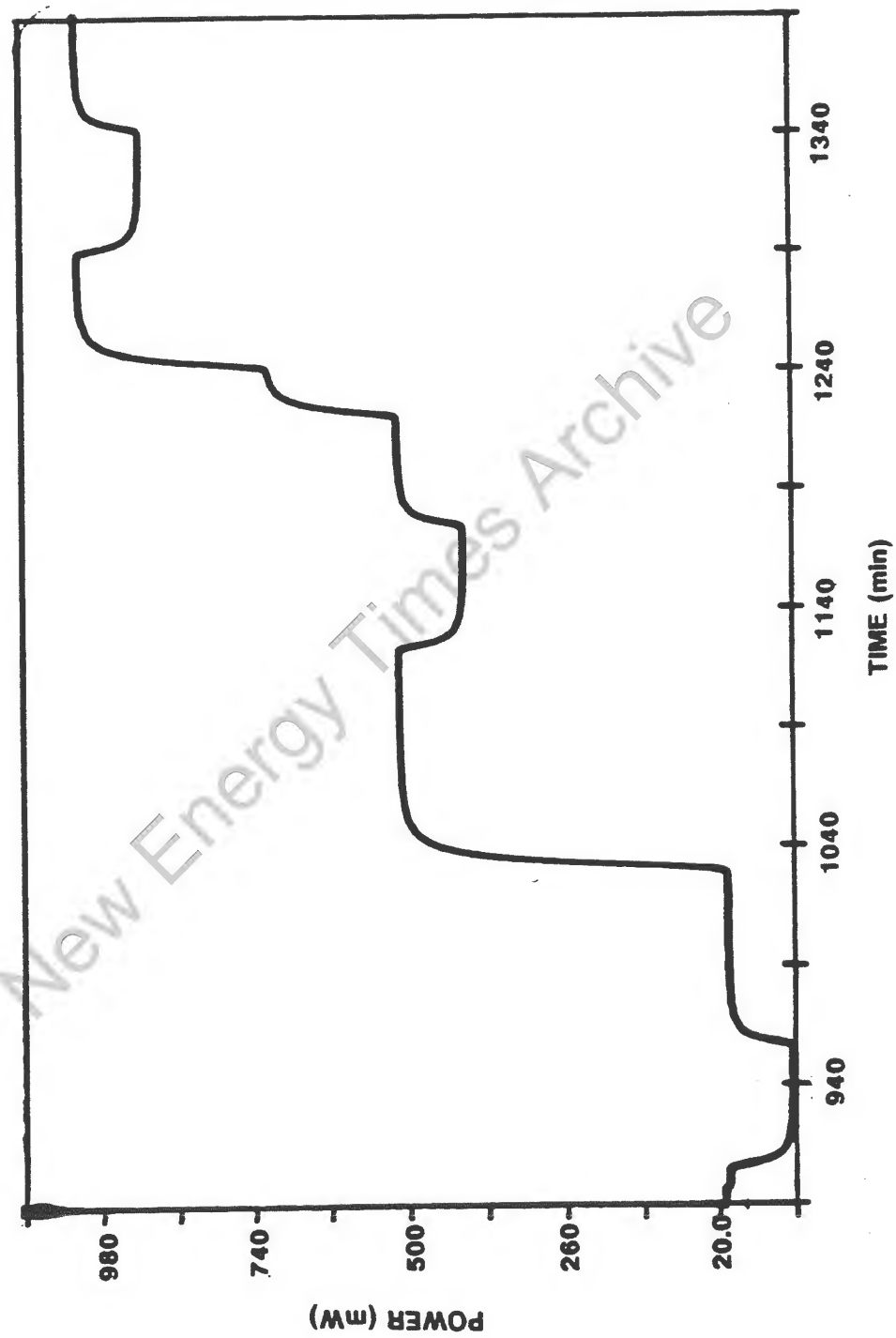
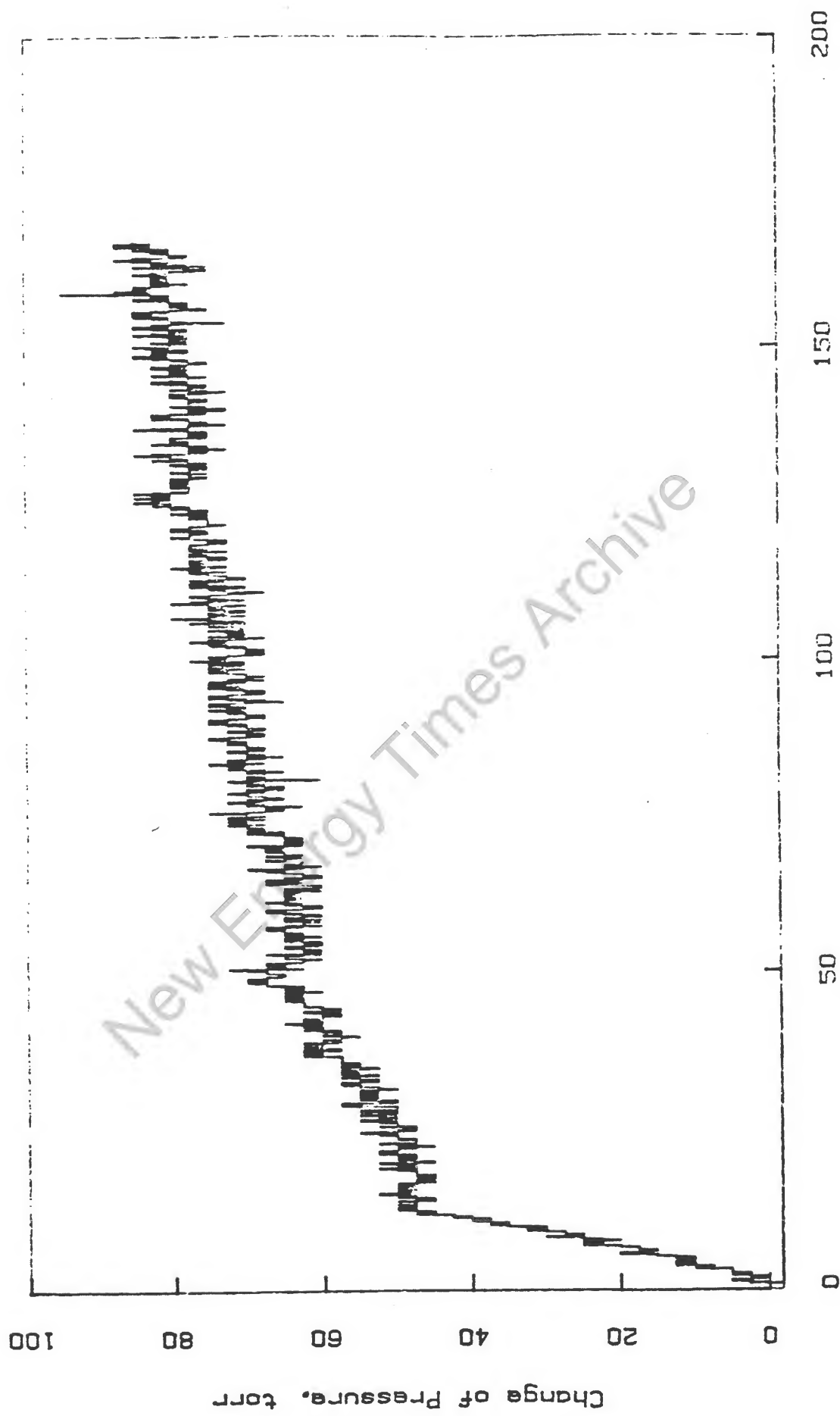


Figure 5

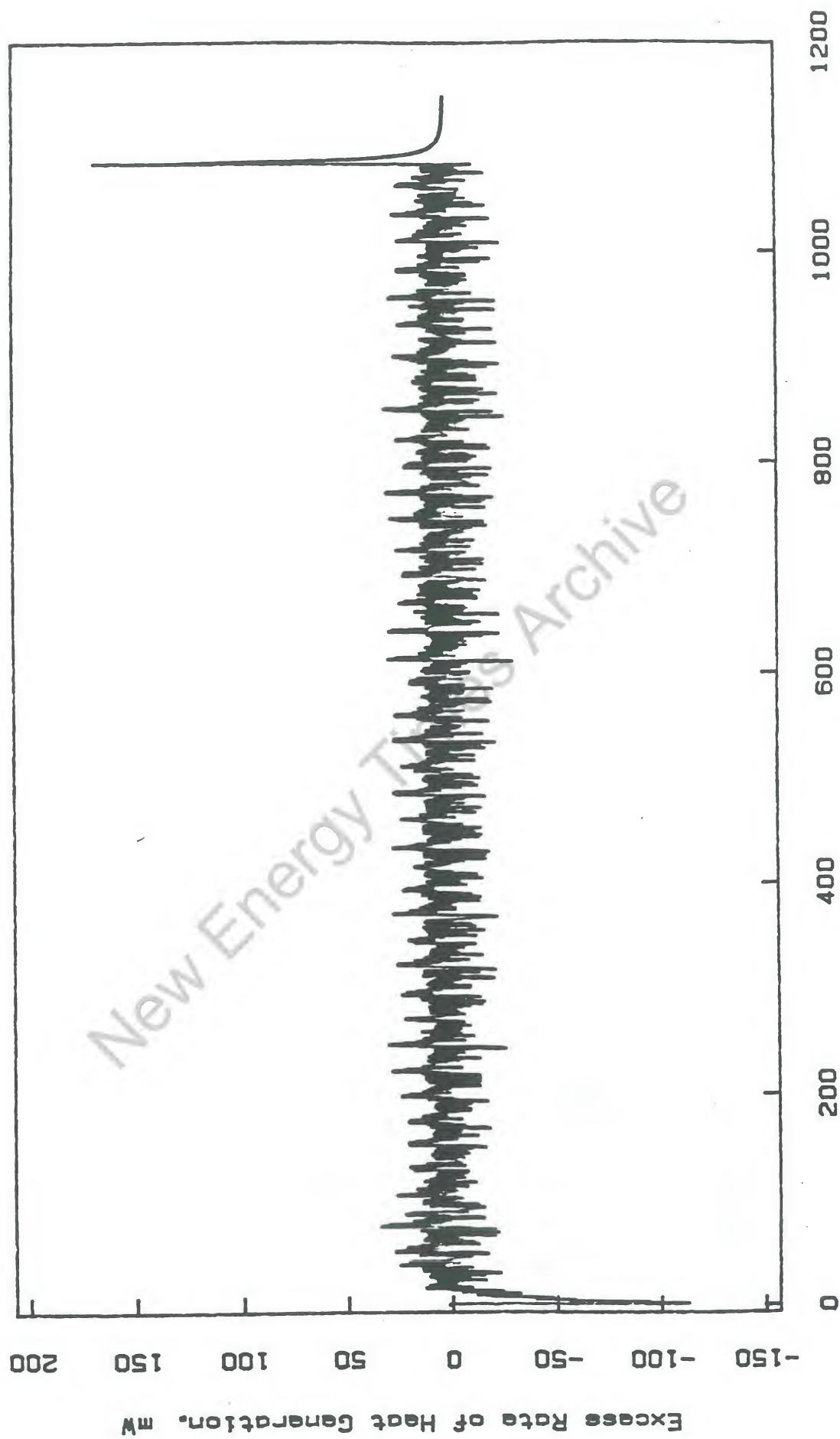
recombination catalyst pressure expt.



Time, minutes

Figure 6

Pt: 0.1M LiOD/D2O; 100 mA/cm²



Time, minutes

Figure 7

Pt: 0.1M LiOD/D2O: 100 mA/cm²

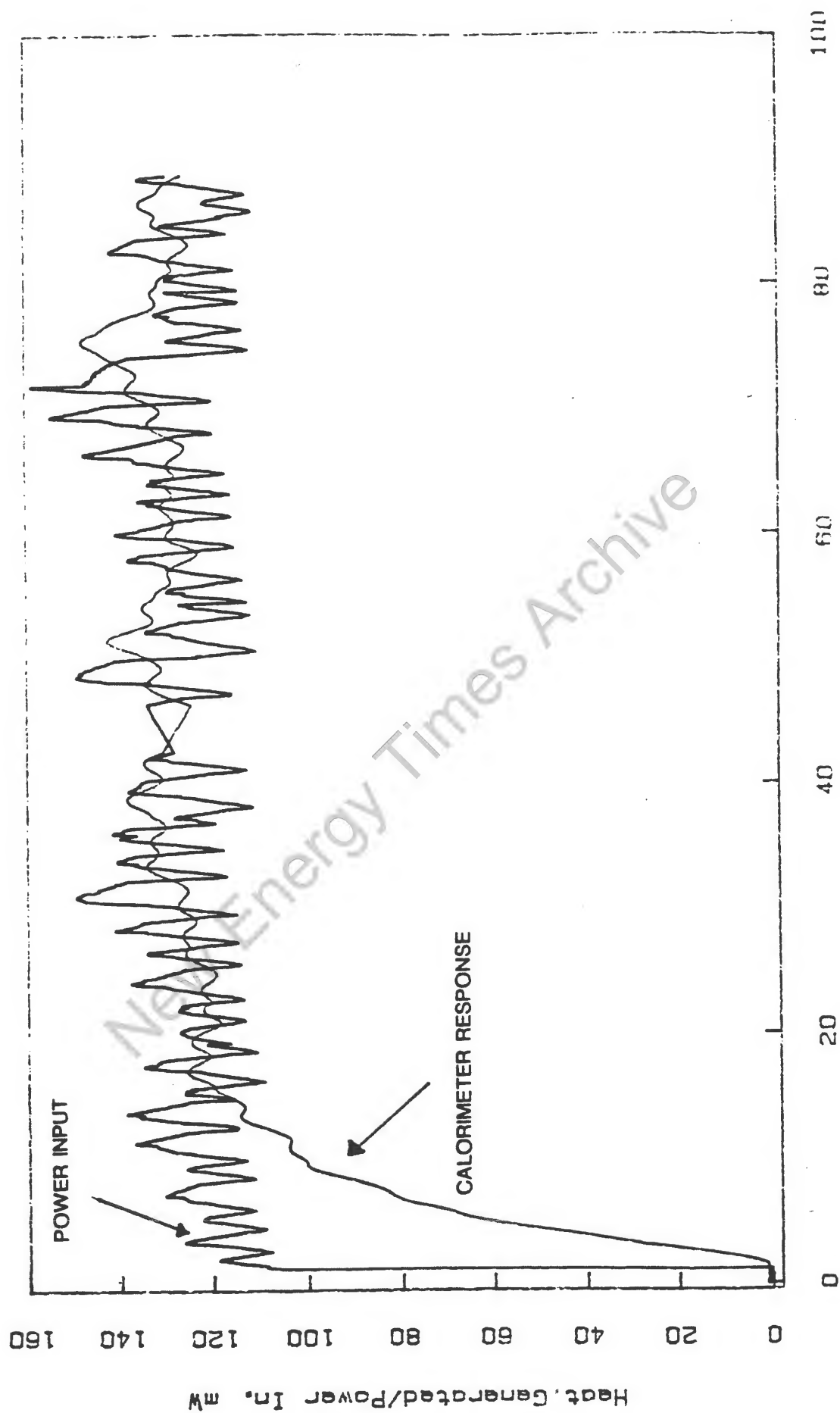
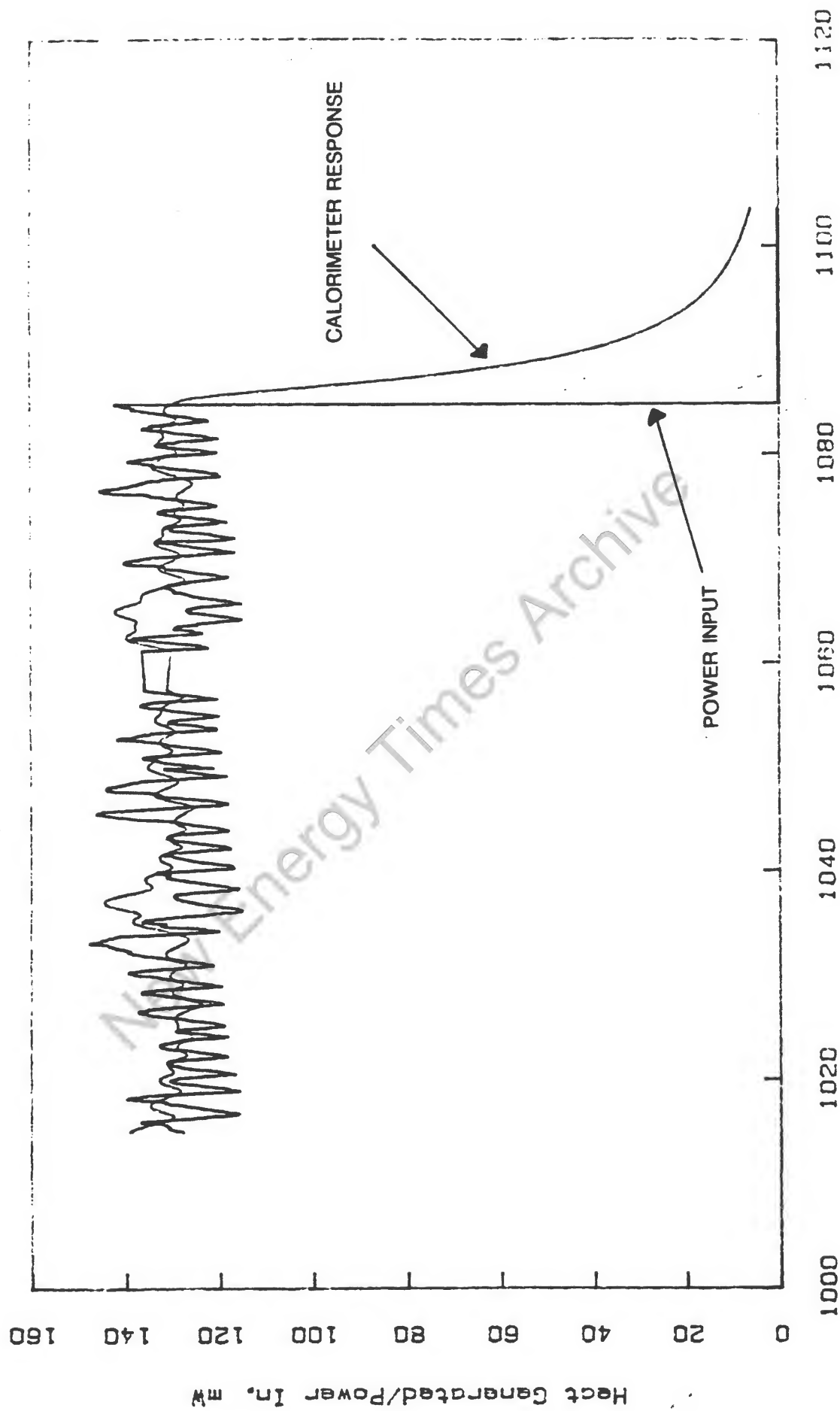


Figure 8

Pt: 0.1M LiOD/D2O; 100 mA/cm²



Time. minutes

Figure 9

2 mm Cast Pd; LiOD; 1100 mJ/s

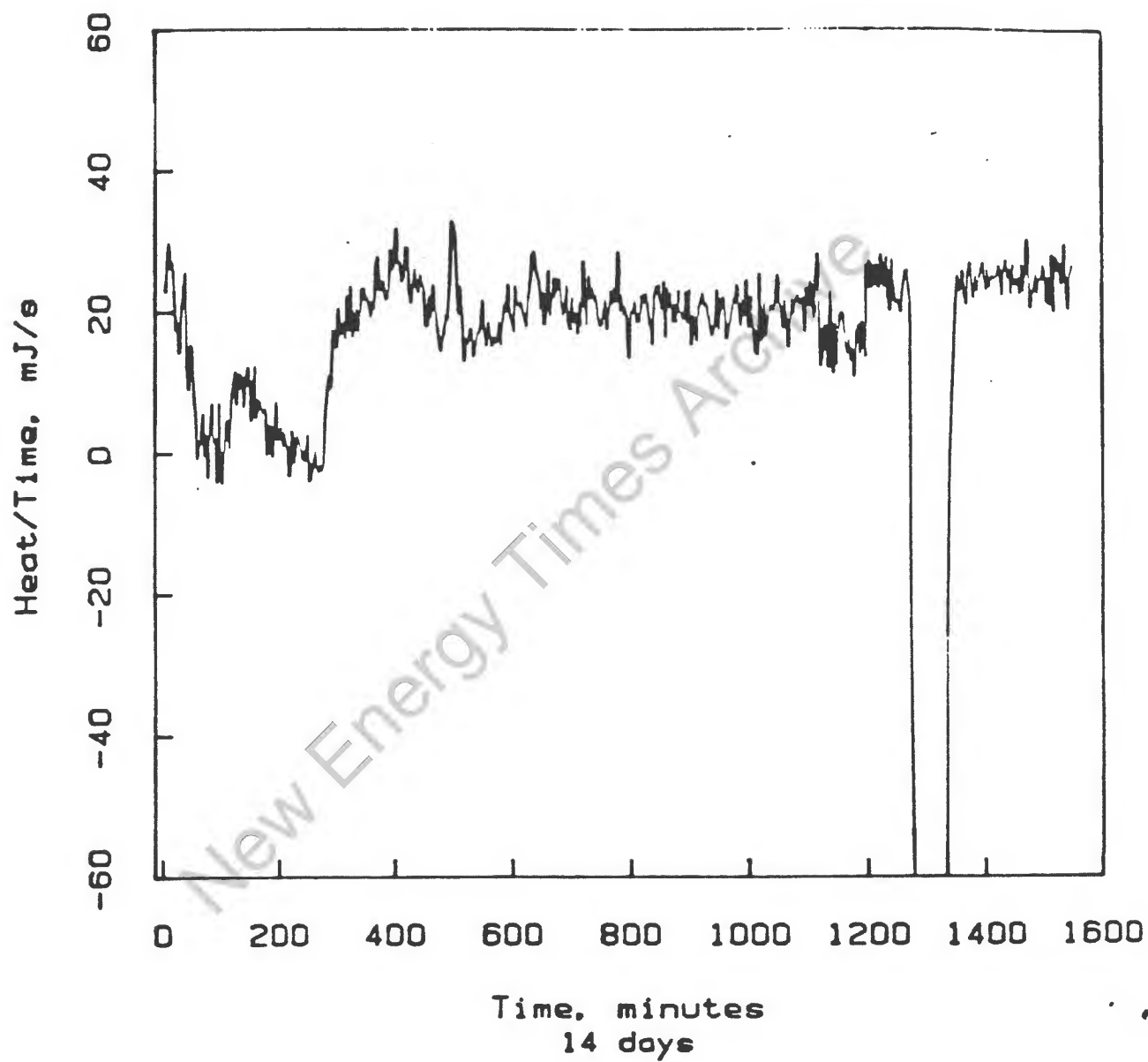


Figure 10

2 mm Cast Pd; LiOD; 1300 mJ/s

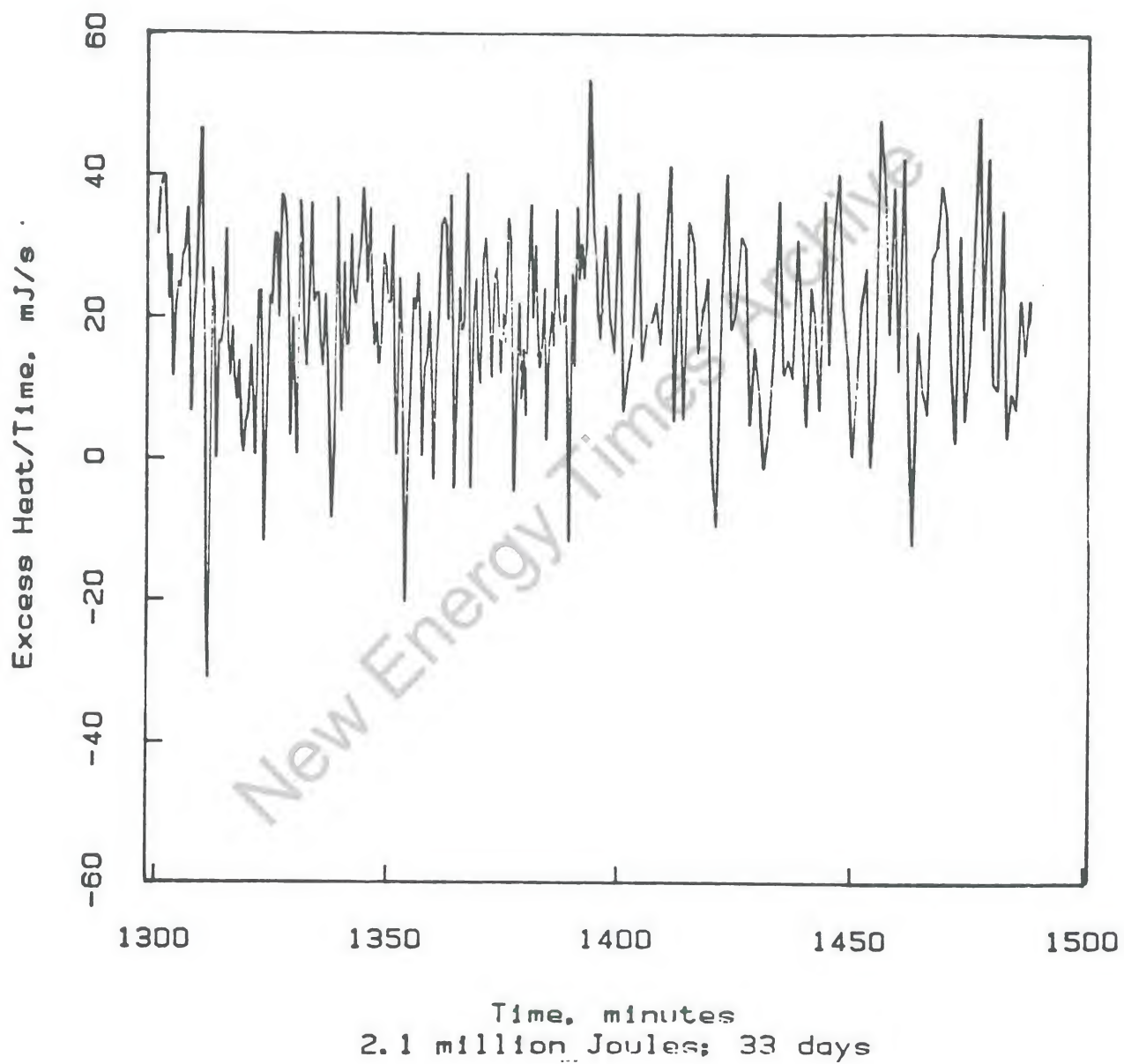


Figure 11

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